Tracing the origin of raw materials used for the production of ancient ceramics: a case study of multi-period archaeological sites in the Turopolje area (Continental Croatia)

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Abstract

The selection of raw materials for ancient pottery production was influenced by a variety of technological and cultural factors, underscoring the importance of characterising these materials to understand the technology of ancient societies. This research examines ancient ceramics from two multi-period archaeological sites: Staro Čiče-Gradišće (Neolithic, Copper Age, Copper/Early Bronze Age, Early Bronze Age, Late Bronze Age, Roman period, and Late Mediaeval period) and Kurilovec-Belinščica (Early Bronze Age and Middle/Late Bronze Age), alongside potential clayey raw materials collected near these settlements. Using a multi-analytical approach (optical microscopy, X-ray powder diffraction, mass and emission spectrometry, and laser granulometry), the research aims to determine the characteristics of the paste recipes (raw clay and tempers) and to examine the type and provenience of the raw materials used for ancient pottery production over different periods of the past (from the Neolithic to the Late Mediaeval period). The results showed that ancient potters preferred to use moderately plastic, sandy clay, while Bronze Age potters often used highly plastic clay. Potters utilised various non-plastic tempering materials, such as sands and gravels, grog, and mollusc shells, with their choice being influenced by the need to enhance technological properties as well as by regional and culturally determined pottery traditions. Most of the ceramics are of local origin, made from easily available raw materials that represent flood sediments of the nearby Sava River. However, non-local materials were detected in Neolithic samples, indicating the presence of exchange networks among those communities.

Keywords: Pottery, raw materials, archaeometry,

1. INTRODUCTION

provenience

The production process for each ceramic object starts with the selection of suitable raw materials. That procedure is very complex and commonly influenced by various cultural factors rather than it is just being a technologically determined choice (RICE, 1987). In addition, raw materials (clayey and tempering material), as the essential elements of each ceramic product, directly affect the production process and properties of the final product (RICE, 1987; VELDE & DRUC, 1999). Their examination is therefore of great importance for understanding the technology of ancient societies, especially in the context of one of the oldest crafts in human history, pottery. From a materials science perspective, the properties of raw materials in ceramic production and the methods of processing result in a specific artefact structure and chemical composition (TITE, 1999). From an archaeological perspective, these properties are indicative of a particular technological tradition, or material culture integral to past societies. The research approach, encompassing ancient materials science, facilitates the investigation of pottery technologies and enhances understanding of the selection of specific raw materials. Simultaneously, the results can offer further insights into the use, exchange, and distribution of these products (TITE, 1999; SILLAR & TITE, 2000).

Following this approach, this paper aims to provide a detailed study of the textural, compositional and technological properties of the raw materials potentially used for ceramic vessel production over a long period of time. Spatially, the research is conducted in the area of Turopolje, near the Sava River, which is located in the southwestern edge of the Pannonian Plain, in Croatia. It is a strategically important, but often neglected border area between two geographical and geological zones, a frequent contact zone of different sociocultural traditions in the past. For these reasons, the regionally significant archaeological site Staro Čiče-Gradišće was chosen as a main case study. Since it is a multi-period site (Neolithic, Copper Age, Copper/Early Bronze Age, Early Bronze Age, Late Bronze Age, Roman period, and Late Mediaeval period), the research questions are focused on the characterization of ancient recipes, determining the differences in type and processing of the raw material used for pottery making between the fifth millennium BC and the first half of the second millennium AD.

To appropriately discuss the selection of raw materials and the production and exchange of ceramics within past communities, it is necessary to collect potential clayey materials from the surrounding area alongside analysing the archaeological ceramics. This approach enables the investigation of the origin and availability of resources, indirectly shedding light on the interactions between past communities and their immediate environment.

In contrast to the numerous studies focused on the provenance of the pottery raw materials (e.g. MARITAN et al., 2005; IORDANIDIS et al., 2009; PAPACHRISTODOULOU et al., 2010; CARLONI et al., 2021; SPATARO, 2011; IONESCU et al., 2011; RATTO et al., 2015; SANTOS RODRIGUES et al., 2015; AMICONE et al., 2020; STERBA et al., 2020; MARITAN et al., 2021), fewer are conducted on both the ceramics and the raw materials collected in the nearby environment (e.g. ŽIBRAT GAŠPARIČ, 2004; BELFIORE et al., 2014; ANDALORO et al., 2011; MICHELAKI et al., 2014; ŠARIĆ et al., 2018; GONZÁLEZ et al., 2018; KAŁASKA et al., 2020; BELTRAME et al., 2021). In Croatia, such research is still very rare (MANGE & BEZECZKY, 2006; ŠEGVIĆ et al., 2012; MARITAN et al., 2019; MIŠE et al., 2020; MIŠE & QUINN, 2022) whereas only several studies examine the potential raw materials collected near the place of production (KUDELIĆ et al., 2018; MIŠE et al., 2019). These studies mainly consider a single archaeological site or specific period, whereas only one recent study provided an insight into the raw materials preferences throughout different periods of the past (NERAL et al., 2023).

To that extent, this research represents a significant step forward in studying the variability of ancient potters' recipes, focusing on multi-period archaeological sites, which will enable the monitoring of changes in the use of resources within the immediate landscape. The study will therefore seek to answer the following questions: Was pottery production exclusively local during all periods of the past? Why were specific raw materials chosen? Was the choice influenced by the availability and characteristics of the immediate environment, the optimisation of technological solutions, or cultural factors? To answer these questions, a multi-analytical approach, including optical microscopy, X-ray powder diffraction, mass and emission spectrometry, and laser granulometry, was applied to both the ceramics and clayey samples. Among these, the optical microscopy of thin sections represents an essential method for determining the ceramic fabric groups and characterizing the potential raw materials, by making fired clay briquettes of the clayey material collected in the vicinity of the sites. In addition, geochemical analysis provides detailed compositional patterns of major, minor, trace and rare earth elements (REE) that enable identification of the specific raw material sources and facilitate differentiation between locally produced and imported ceramics. Therefore, to enhance the understanding of raw material selection processes and ensure a more robust dataset for considering their provenance and distribution over time, the research incorporates geochemical data from ceramic sherds and clay samples from the nearby Kurilovec-Belinščica site (Early Bronze Age and Middle/Late Bronze Age). Accordingly, this study presents the results of integrated mineralogical, petrographic, and chemical analyses of various ceramic assemblages from multi-period archaeological sites, alongside clayey sediments gathered in their proximity.

2. GEOLOGICAL AND ARCHAEOLOGICAL SETTING

2.1. Geological setting

The Turopolje region is situated in the central part of the Republic of Croatia (Fig. 1). The area of approximately 600

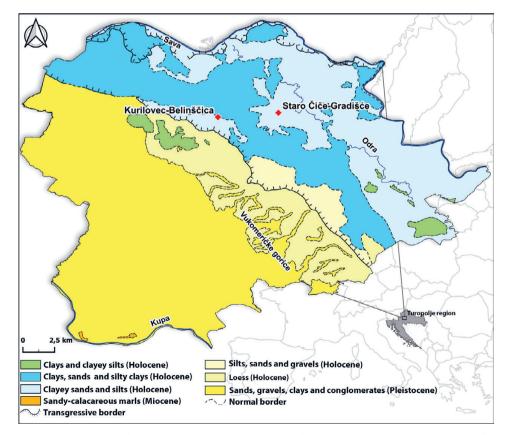


Figure 1. The location and geological map of the Turopolje region with locations of investigated archaeological sites (modified after: PIKIJA, 1987b; BASCH, 1981; KUDELIĆ & SIROVICA, 2022).

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km² is bordered by the two rivers: the Sava in the northern and the Kupa in the southern part. Geologically, the Turopolje is situated in the southwestern part of the Pannonian Basin System (PBS), a depositional area that palaeogeographically belonged to the Central Paratethys (RÖGL & STEININGER, 1984; PAVELIĆ, 2001; KUREČIĆ, 2017). The relief of Turopolje is represented by two main morphographic types: the Sava River valley and Vukomeričke gorice, the hilly relief of smaller horsts formed by uplift along the Dinaric extension fault (VELIĆ, 1983). The most common and the oldest deposits that outcrop along the horsts are the Viviparus beds composed mostly of sand, gravel and clay sediments of Pliocene age. The youngest, Plio-Quaternary fluvial-lacustrine sediments composed of the alternation of gravel, sand and clay deposits and Pleistocene loess sediments are less frequent (ŠIKIĆ et al., 1978, 1979; PIKIJA, 1987a, 1987b; KUREČIĆ, 2017). The alluvial Sava River valley consists mainly of Quaternary deposits, and is divided into three lithostratigraphic units. The oldest lithostratigraphic unit comprises gravels, sands, and clays whereas the middle one consists of loess deposits, fine clayey silts with interlayers of sands and gravels and, sporadically, peat and swamp sediments. The youngest unit is composed of gravels, sands, and silty clays (ŠIMUNIĆ & BASCH, 1975; ŠIKIĆ et al., 1978, 1979; BASCH, 1983; PIKIJA, 1987a, 1987b; BARUDŽIJA et al., 2020). The multiperiod archaeological sites Staro Čiče-Gradišće and Kurilovec-Belinščica are situated on fine-grained, floodplain sediments of the Sava River formed as the result of occasional floods occurring in the wider area of the valley. The thickness of floodplain sediments in the longitudinal profile increases from west to east, while in the transverse profile, it decreases as it approaches the Sava riverbed. Flood sediments consist of finegrained unconsolidated deposits like gravels, sands, sandyclays or clayey silts with a transition to silty clays (BASCH, 1981, 1983). These deposits are mainly of carbonate origin, composed of limestone and dolomite, while quartz, siliciclastic sediments and fragments of siliciclastic rocks are subordinate (CRNKOVIĆ & BUŠIĆ, 1970; BARUDŽIJA et al., 2020).

2.2. Archaeological setting

The Turopolje is a cultural and historical region situated on the south-western fringe of the Pannonian Plain. It is primarily a marshy area located directly along the course of the Sava River on the edge of the contact zone with the Adriatic or Karst region. This strategically important area and the crossroads of communication routes were the reason for the establishment of the settlement in not always ideal living conditions.

This paper presents the ceramic assemblages from two archaeological sites. Staro Čiče-Gradišće is one of the sites in the lowland of Turopolje (Fig. 1) and was almost continuously inhabited from the Neolithic to the late Middle Ages. This makes this archaeological site unique in the wider area of continental Croatia and the only site of the tell type. The archaeological excavations conducted back in the 80s (DURMAN, 1983; TEŽAK-GREGL & VOJVODA, 1987) brought to light a stratified sequence with repeated layers of occupation phases dated to the Neolithic, Cooper Age, Early and Late phase of the Bronze Age (BALEN-LETUNIĆ, 1996; KUDELIĆ & SIROVICA, 2022), Late Antiquity and the Late Medieval period.

In contrast, the nearby Kurilovec-Belinščica site (Fig. 1) is a multi-period, primarily Bronze Age site with a sporadic occurrence of Roman-period and modern-period archaeological remains. The most intensive activities were recorded during the Middle and early phases of the Late Bronze Age, with settlement remains dated to between the 15th and 13th centuries BC (KUDELIĆ, 2016; KUDELIĆ & SIROVICA, 2022). However, modest remains and very fragmented pottery dated to the Early Bronze Age period between the middle of the 22nd to the middle of the 20th century BC were also discovered at the site (KUDELIĆ & SIROVICA, 2022).

The positions of these sites were strategic, and probably played a key role, especially the site of Staro Čiče-Gradišće, in the cultural and economic dynamics between the population in the border area between Pannonia, the Alps and the eastern Adriatic.

3. MATERIALS AND METHODS

3.1. Archaeological ceramics

Investigating pottery from the site of Staro Čiče-Gradišće, over 2,000 fragments, mostly prehistoric, were examined macroscopically. Based on macroscopically determined groups of ceramic fabrics, 40 representative samples were selected for archaeometric analysis covering all the periods represented (Table 1, Fig. 2). To obtain a more reliable geochemical comparison of ceramics from the Turopolje region, the research includes geochemical data of ceramic sherds from the Bronze Age site Kurilovec-Belinščica (14 samples, Table 1, Fig. 2) the mineralogical and petrographic characteristics of which were previously published in KUDELIĆ et al. (2018).

3.2. Clayey samples

Field sampling of potential clayey raw materials was conducted in the vicinity of the Staro Čiče-Gradišće site and was focused on watercourses that are common sources of clayey sediments. Samples were collected in a zone up to 500 m away from the archaeological site. A total of 10 samples were collected in seven locations using a hand auger from 0.5 to 1.4 m in depth. In order to obtain a more reliable data set of geochemical compositions, the research also includes three clayey samples collected in the vicinity of the Kurilovec-Belinščica site, the mineralogical and petrographic characteristics of which were previously published (KUDELIĆ et al., 2018).

3.3. Analytical methods

To examine paste recipes and the characteristics and provenance of the raw materials used in ancient pottery production, the ceramic and clayey samples were analysed using the following methods: ceramic petrography, X-ray powder diffraction (XRPD), inductively coupled plasma- mass spectrometry (ICP-MS) and emission spectrometry (ICP-ES) and laser granulometry. Analytical methods were conducted at the University of Zagreb, Faculty of Mining, Geology and Petroleum Engineering and at the Institute of Archaeology in Zagreb.

	Sample number	Vessel type	Period	ОМ	XRPD	ICP-ES, ICP-MS
	252	pot	Late Mediaeval	+	-	-
	253	pot	Late Mediaeval	+	-	+
	254	pot	Late Mediaeval	+	-	-
	255	pot	Late Mediaeval	+	+	-
	256	pot	Late Mediaeval	+	+	+
	257	pot	Late Mediaeval	+	-	-
	258	lid	Roman period	+	-	+
	259	/	Roman period	+	+	-
	260	/	Roman period	+	+	-
	261	pot	Late Bronze Age	+	-	+
	262	pot	Late Bronze Age	+	+	-
	263	pot	Early Bronze Age	+	+	-
	264	pot	Early Bronze Age	+	-	+
	265	/	Early Bronze Age	+	-	+
	266	/	Early Bronze Age	+	-	-
	267	/	Early Bronze Age	+	+	-
	268	/	Early Bronze Age	+	-	-
, Ç	269	/	Copper/Early Bronze Age	+	+	-
adiš	270	/	Copper/Early Bronze Age	+	-	-
Ģ	271	bowl	Copper/Early Bronze Age	+	-	+
Staro Čiče-Gradišće	272	/	Copper/Early Bronze Age	+	-	-
õ	273	bowl	Copper Age	+	-	-
Staı	274	bowl	Copper Age	+	+	-
	275	/	Copper Age	+	_	-
	276	,	Copper Age	+	_	-
	270	/	Copper Age	+	-	+
	278	/	Copper Age	+	_	+
	270	/	Copper Age	+	+	-
	280	/	Copper Age		т	-
	280	/	Copper Age	+	-	+
	282	/	Copper Age	++	+	-
	283		Neolithic	+	+	<u> </u>
	284		Neolithic		т	
		pot		+	-	+
	285	pot	Neolithic	+	+	+
	286	/	Neolithic	+	-	+
	287	/	Neolithic	+	-	-
	288	pot	Neolithic	+	+	-
	289	pot	Neolithic	+	+	-
	290	/	Neolithic	+	-	-
	291	/	Neolithic	+	+	-
	14130	bowl	Middle/Late Bronze Age	-	-	+
	14131	pot	Middle/Late Bronze Age	-	-	+
	14134	small jug	Middle/Late Bronze Age	-	-	+
a	14135	/	Middle/Late Bronze Age	-	-	+
ščic	14138	/	Middle/Late Bronze Age	-	-	+
Kurilovec-Belinščica	14139	tripod pan	Middle/Late Bronze Age	-	-	+
vec	14144	bowl	Middle/Late Bronze Age	-	-	+
rilo	14146	bowl	Middle/Late Bronze Age	-	-	+
Ku	14147	bowl	Middle/Late Bronze Age	-	-	+
	14148	bowl	Middle/Late Bronze Age	-	-	+
	14150	bowl	Middle/Late Bronze Age	-	-	+
	14151	pot	Middle/Late Bronze Age	-	-	+
	14152	pot	Middle/Late Bronze Age	-	-	+
	14223	/	Early Bronze Age	_	_	+
	. 1223	/	Lang Dionze Age			

The clayey samples were moulded in small briquettes, dried at room temperature and fired in a laboratory kiln at 650 °C for one hour with a 30-minute soaking period to investigate the characteristics of the collected clayey samples and obtain a relevant comparison with clayey materials found in archaeological ceramics. Sample 101 was unsuitable for modelling clayey briquette due to its high sand content, which prevents proper briquette formation. The plasticity of the clayey samples was measured on-site, by simulating the conditions of actual processing and by manual modelling of a clay roller and observing deformation by bending force (plasticity range: no plasticity, low, moderate, and high plasticity).

Ceramic sherds and fired clay briquettes were cut vertically and prepared as thin sections for optical microscopy. The samples were studied under a polarizing microscope Zeiss Axiolab 5 in transmitted light. The petrographic analysis of ceramics included the determination of the colour of the matrix, its texture, optical activity, mineral composition, presence of natural and anthropogenic inclusions, voids, and pores. The fabric groups were determined according to the mineralogical composition, amount, size and sorting of the inclusions in the matrix (PCRG, 2010; QUINN, 2013) which indicates different types of clayey raw materials. Fabric subgroups were determined according to the type, size, amount, roundness and bi- or polymodal grain size distribution indicating temper materials.

For the X-ray powder diffraction (XRPD, Table 1), approximately five grams of the archaeological ceramics and clayey samples were crushed to a powder fraction using Retsch vibratory disc mill RS 200. Analysis was conducted on a Panalytical Empyrean diffractometer equipped with Cu-Ka radiation. Scan settings were $3-70^{\circ}2\theta$ on the rotating sample stage, 0.013 °20 step size, 80 s per step. Mineral identification was conducted according to MOORE & REYNOLDS (1997) using PANalytical X'Pert HighScore software with standardized Powder Diffraction Files of the International Centre for Diffraction Data (ICDD). The semiguantitative mineralogical composition was estimated using the Reference Intensity Ratio (RIR) method (SNYDER, 1992), data obtained by calcimetry and diffraction maxima of the reference materials (quartz). In order to determine the type of the clay minerals, the $< 2 \mu m$ fraction of each clayey sample was isolated from the bulk samples and recorded after the following treatments: K and Mg saturation, ethylene glycol solvation, dimethyl sulfoxide solvation, heating to 350 °C and 650 °C.

Grain size distribution (in volumetric fractions) of clayey samples was estimated using a laser granulometer Malvern Panalytical Mastersizer 3000 Particle Size Analyzer with Hydro EV unit and < 5 % obscuration. Due to the volumetric fraction measurement and non-spherical particle shapes, the results of this method are not comparable to those based on mass fractions.

To examine the shrinkage, 50 g of each clayey sample collected on site was modelled in a roller approximately 7 x 2 cm in size and left to dry at room temperature. The length and diameter of each roller were measured until they were completely dry. Relative shrinkage (%) was assessed by the

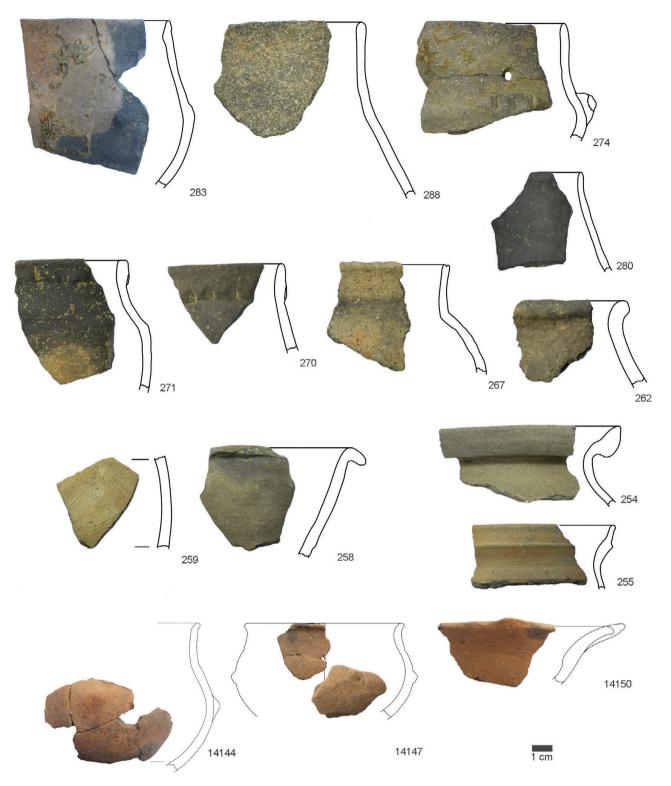


Figure 2. Representative pottery from the Staro Čiče-Gradišće and Kurilovec-Belinščica sites. More information about the ceramics is given in Table 1.

difference between the original and dried volumes of each sample.

A selection of 27 ceramic sherds (13 from Staro Čiče-Gradišće and 14 from Kurilovec-Belinščica) and five clayey samples (two from Staro Čiče-Gradišće and three collected near Kurilovec-Belinščica site) were subjected to inductively coupled plasma- mass (ICP-MS) and emission spectrometry (ICP-ES) in the Bureau Veritas Minerals laboratory. The concentration of major oxides, together with minor elements, was determined by ICP-ES after lithium borate fusion, whereas the composition of trace and rare earth elements (REE) was determined after modified *aqua regia* digestion (1:1:1 HNO₃:HCl:H₂O) by ICP-MS. The detection limit for major oxides; SiO₂, Al₂O₃, MgO, CaO, Na₂O, K₂O, TiO₂, P₂O₅ is 0.01 %, for Cr₂O₃ the limit is 0.002 % and for Fe₂O₃ is 0.04 %. Detection limits for minor and trace elements are: V=8 ppm; Ba, Zr, Nb=5 ppm; Y=3 ppm; Sr=2 ppm; Sc, Be, Sn, Zn=1 ppm; Ga, Sr, W, Au, Se=0.5 ppm; Nd=0.3 ppm; Co,

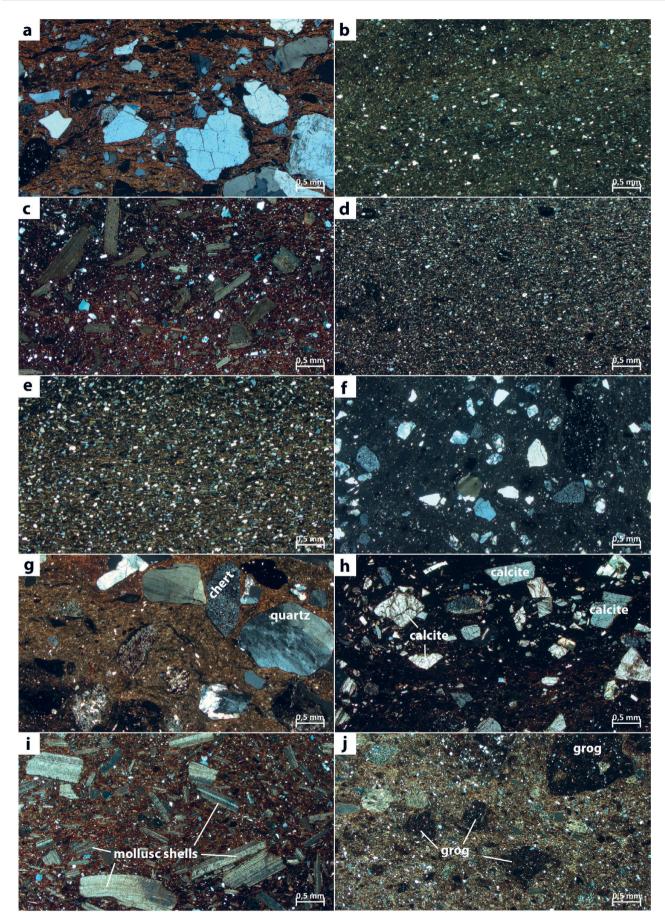


Figure 3. Thin section photomicrographs of ceramics from the Staro Čiče-Gradišće site. (a) Fabric group 1, sample 291, (b) fabric group 2, sample 285, (c) fabric group 3, sample 279, (d) fabric group 4, sample 283, (e) fabric group 5, sample 286, (f) unclassified overfired sample 254, (g) lithoclast tempered ceramic sample 264, (h) calcite tempered sample 288, (i) mollusc shells tempered sample 274, (j) grog tempered sample 282. All photographs are taken in cross polarized light (XPL).

Th=0.2 ppm; Cs, Hf, Rb, Nb, Ta, U, La, Ce, Mo, Cu, Pb, Ni, Cd, Sb, Ag, Tl=0.1 ppm; Gd, Dy, Yb=0.05 ppm; Er=0.03 ppm; Pr, Eu, Ho=0.02 ppm; Tb, Tm, Lu, Hg= 0.01 ppm. The results of geochemical analyses were subjected to bi- and multivariate statistical analysis in TIBCO Statistica Software 2020. All the elements with concentrations below the detection limit were excluded from the statistical analysis.

4. RESULTS

4.1. Ceramic petrography

The ceramic matrix is composed of naturally present, very fine (< 0.1 mm) and fine (0.1 - 0.25 mm) quartz, feldspar, and mica (muscovite and biotite) crystalloclast inclusions that are sparsely (< 10 %) to abundantly (40 %) present. These crystalloclasts are sub-angular and well to very well distributed in the matrix. By observing the amount and size of mineral inclusions in the clay matrix, five fabric groups have been determined (Fig. 3, Table 2). The matrix colour is brown to dark brown in PPL (plane polarized light) while the optical activity of the matrix, in XPL (cross polarized light), varies from low to high but it is mostly medium-high. Two samples (254 and 257) remain unclassified regarding the highly vitrified clay matrix (Fig. 3, Table 2).

Along with naturally present crystalloclasts in the matrix, fragments of sedimentary, metamorphic, and igneous rocks (lithoclasts) are present in 18 samples (45 % of total samples, Fig. 3g). Sedimentary rocks are the most abundant within the first fabric group (1). These are mostly siltstones whereas limestone, dolomite and chert are less frequent. Igneous rocks are present in most of the samples and are composed of feldspar, biotite and quartz indicating their acidic origin or quartz bearing intermediate rocks. Among metamorphic rocks, quartzite is the most common, whereas schist is rarely

present. Most of these lithoclasts-tempered samples contain mono- and polycrystalline quartz which sometimes contain cracks. Lithoclasts are moderately to very commonly present (7-30%), fine to very coarse (0.1 -> 3 mm) in size and mostly poorly to moderately sorted in the matrix. Most of the samples contain both sub-angular and sub-rounded lithoclasts although the sedimentary inclusions are more rounded than the igneous and metamorphic clasts. Very angular, fine to very coarse and poorly sorted calcite is recorded in one sample (288, Fig. 3h). Besides, nine samples (23 %) contain moderate to very common (10 - 30%) amounts of elongated and slightly curved mollusc shell fragments (Fig. 3i). Grog (cheramoclasts) is determined in 12 samples (30 %, Fig. 3j). The composition of grog is most often similar to the composition of the ceramic matrix whereas the main difference is in the colour and, sporadically, in the amount of very fine and/or fine crystalloclast inclusions. The amount, shape, and bimodal grain size distribution suggest that lithoclasts, calcite, grog, and mollusc shells were intentionally added inclusions (tempering material). Therefore, by observing these intentionally added inclusions (tempering material), 14 fabric subgroups have been determined. Clay pellets were recorded in negligible quantities. Most of them are transparent with sharp and clear edges. The amount, size, and shape of the pores vary significantly among the samples. The pores are moderately present, 10 % on average, mostly elongated, parallel to the vessel walls, and sporadically filled with secondary calcite. Rounded and irregular pores are less frequent.

The mineral composition of the fired clay briquettes is very similar in all the samples, composed of angular and subangular monocrystalline quartz and feldspar inclusions (Fig. 4, Table 3). Among the mica minerals, muscovite is the most common whereas biotite occurs less frequently. Most of the samples contain sparse amounts (3 - 7 %) of fine and sub-

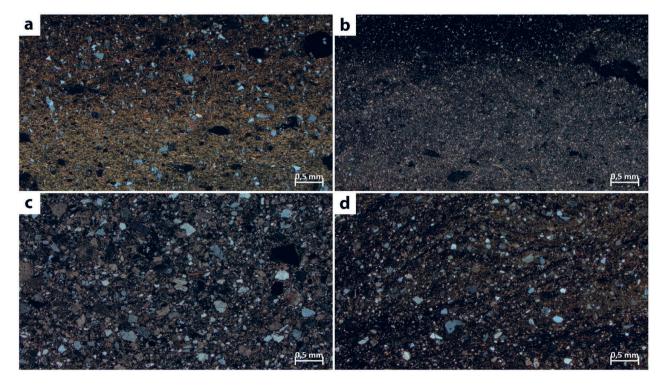


Figure 4. Thin section photomicrographs of fired clay briquettes; (a) sample 102, (b) sample 105, (c) sample 107 and (d) sample 108.

Table 2. Ceramic petrography analysis of pottery thin sections from the Staro Čiče-Gradišće site (L – low, MH – medium-high, H – high; F – fine, M – medium, C – coarse, VC – very coarse, PS – poorly sorted, MS – medium sorted, WS – well sorted, VWS – very well sorted, SA – sub-angular, VA – very angular, SR – sub-rounded, + – present, ++ – dominant, / – unable to estimate the amount of crystalloclast in the matrix since the samples are overfired.

		Fabric squogroups	1.1	1.1	1.1	1.1	1.1	1.1	1.2	1.3	1.3	1.1	1.1	1.1	2.1	2.2	2.3	2.3	2.3	2.4	2.3	2.4	2.5	2.6	3.1	 	2.0	3.2	3.1	3.1	3.1	3.1	3.2	4.1	4.2	4.2	4.2	4.1	г 1	
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		Temper size	F/C	F/C	VWS	F/M	M/C	M/C		Σ	F/VC	M/C	F/C	F/C	M/C	M/VC	F/C	F/C	F/C	F/M	F/C	F/M	T	F/VC	·	-		F/C	: '	ı	ı	,	F/C	F/M	,	·	·		,	
		Temper (%) Tunoms	30	25	30	40	20	15	! .	7	30	30	40	30	25	20	30	30	20	10	20	15	I.	25	ı	, c	75 15	25	¦ '	,	,	,	30	15						
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		Feldspar	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+ -	+ -	+ +	- +	• +	+	+	+	+	+	+	+	+	+	+	-
	Raw clay	Quartz	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+ -	+ +	- +	+	+	+	+	+	+	+	+	+	+	+	-
	Ř	Fine crystalloclasts (%)							ı										2 10										10 – 15							3 – 10			15	2
		Very fine crystalloclasts (%)						:	<10										1E JE										20 – 30 1							40			40	2
vernreg.		Optical activity Very fine	т	HM	HМ	т	MH	HM	Т	н	т	т	н	н	MH	HM	т	MH			НМ	НМ	HW	MH	Ξ	HIM	HW			НМ	MH	HM	т	HM	_	т	НМ	HM	Т	-
samples are overnred		Sample	252	253	255	259	266	268	278	280	281	289	290	291	256	264	273	274	275	276	277	282	285	288	261	707	202	267	269	270	271	272	279	258	260	283	284	287	286	2004

	• • • • • • • • • • • • • • • • • • •
Table 2 Potrographic analysis of clayov sodiments collected in the vicini	y of the Staro Čiče-Gradišće archaeological site (MH – medium-high; + – present).
Table 3. Felloulabilic allaivsis of clavey seulitients collected in the vicini	v of the state cite-diadiste archaeological site (with = inequality), $\tau = $ diesent).

Sample number	Optical activity	Very fine crystallo- clasts (%)	Fine crystallo- clasts (%)	Quartz	Feldspar	Muscovite	Biotite	Metamor- phic and igneous frag- ments	Sedimentary fragments	Mollusc shells
99	MH	50	2	+	+	+	-	-	-	+
100	MH	15	25	+	+	+	-	+	+	+
102	MH	30	10	+	-	+	+	+	+	+
103	MH	30	10	+	+	+	-	+	-	-
104	MH	25	15	+	+	+	-	-	+	-
105	MH	40	3	+	+	+	-	-	+	+
106	MH	20	20	+	+	+	+	-	+	+
107	MH	15	30	+	+	+	-	+	+	+
108	MH	25	15	+	+	+	-	+	-	-

angular to sub-rounded metamorphic (quartzite, schist) and igneous rocks. Sedimentary fragments are siltstones and less commonly dolomites, fine-grained in size and mostly subrounded. Larger quartz grains have cracks as a result of the firing process. According to the high amount of the crystalloclasts in the matrix, all of the samples are determined as sandy clay. However, three samples (100, 106 and 107, Fig. 4) can be discriminated from others regarding their significantly high amount of fine and medium quartz and feldspar inclusions.

4.2. Mineralogy

Quartz, K-feldspar and plagioclase are the major components in almost all of the ceramic samples, partly as a result of the igneous and metamorphic inclusions present in the samples (Table 4). The abundance of calcite discriminates samples 274 and 288 from all others. In sample 274, it suggests the thermal decomposition from aragonite in mollusc shells fragments (shell-tempered ceramics) whereas in sample 288 it reflects the common presence of calcite inclusions determined by optical microscopy. Calcite is commonly associated with the traces of dolomite. Spinel, haematite and anatase/rutile are rarely present in the samples. The diffraction maxima at 10 Å, corresponding to mica and a dehydroxylated illite-like phase is determined in all the samples. The traces of 14 Å phyllosilicates are sporadically recorded (samples 267, 274 and 282).

The mineralogical composition of the clayey samples (Table 4) consists mostly of quartz, K-feldspar, plagioclase and mica, including mica-illite and illite/smectite. Carbonates, including calcite and dolomite, are characteristic of all the samples but the amount of dolomite generally exceeds that of calcite. The traces of ferrous minerals (haematite, goethite and/ or pyrite) are present in most of the samples whereas amphibole is only recorded in sample 105. The treatments conducted on the $< 2 \mu m$ fraction revealed differences in the clay mineral compositions. Samples 102, 103, 104 and 108 (Fig. 5) are

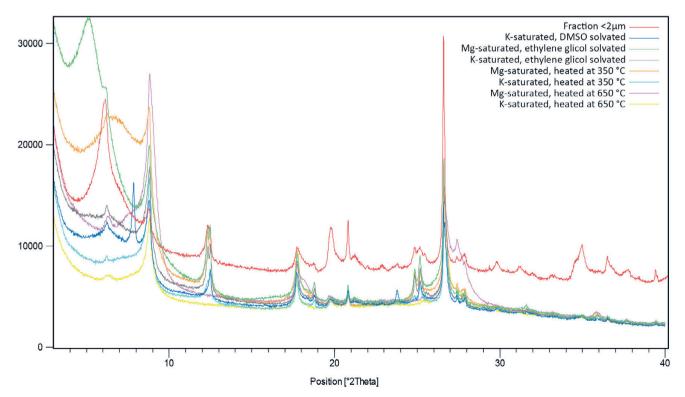


Figure 5. X-ray powder diffraction patterns of clayey sample 108.

Table 4. Semiquantitative mineralogical composition of ceramics from the Staro Čiče-Gradišće site and clayey material collected near the site determined by X-ray powder diffraction (Qtz – quartz, Pl - plagioclase, Kfs – K-feldspar, Mca – micaceous material; including mica, illite and illite/smectite, Cal – calcite, Dol – dolomite, Amph – amphibole, Hem – hematite, Gt – goethite, Sp – spinel, Ant – anatase, Rt – rutile, Kaol – kaolinite, Chl – chlorite, Sm – smectite, Vm – vermiculite - – not detected, * – traces, + – minor, ++ – intermediate, +++ – major).

	Sample number	Qtz	PI	Kfs	Мса	Cal	Dol	Amph	Hem/Gt	Sp	Ant/Rt	Kaol	Chl	Sm, Vm
	255	+	+	+++	+	+	*	-	-	-	-	-	-	-
	256	++	+	+	+	+	*	-	-	-	-	-	-	-
	259	+	+	+	++	+	*	-	-	-	-	-	-	-
	260	+	+	++	+	*	-	-	-	-	-	-	-	-
	262	+	+	+	+	-	*	-	-	-	-	-	-	-
	263	+	++	+	+	+	-	-	-	-	-	-	-	-
s	267	++	+	+	+	*	*	-	-	-	-	*	*	-
Ceramics	269	++	-	+	+	-	-	-	-	*	*	-	-	-
era	274	*	+	-	+	+++	*	-	-	-	-	*	*	-
0	279	+	-	+	+	+	-	-	*	-	-	-	-	-
	282	+	-	+	+	+	-	-	-	-	-	*	*	-
	283	++	+	+	+	-	-	-	-	-	-	-	-	-
	285	+	+	+	++	+	-	-	-	-	-	-	-	-
	288	+	+	-	+	+++	-	-	-	-	-	-	-	-
	289	++	+	+	+	*	-	-	-	-	-	-	-	-
	291	+	+	++	+	*	-	-	-	-	-	-	-	-
	99	++	+	+	++	+	+	-	*	-	-	+	+	*
	100	++	+	+	++	+	+	-	*	-	-	+	+	*
_	101	++	+	+	++	+	+	-	-	-	-	*	*	*
erial	102	++	+	+	++	-	*	-	*	-	-	+	+	++
nati	103	++	+	*	++	-	*	-	*	-	-	+	+	++
Clayey material	104	++	+	+	++	*	*	-	*	-	-	+	+	++
Clay	105	++	+	+	++	*	+	*	-	-	-	+	+	*
0	106	++	+	+	++	+	+	-	-	-	-	+	+	*
	107	+++	+	+	++	*	+	-	-	-	-	++	++	*
	108	++	+	+	++	*	+	-	-	-	-	+	+	++

discriminated from others regarding the high swelling clay mineral content, preferably vermiculite, which corresponds to the highest amount of the clay fraction determined by laser granulometry. The highest content of swelling clays is determined in sample 108. Conversely, the other samples are characterized by kaolinite and chlorite with the trace presence of swelling clays. Mixed-layer clay minerals are less frequently present. The dominance of the 10 Å phyllosilicates (mica) and clay minerals (illite) together with the traces of 7 and 14 Å clay minerals is in line with "no plasticity" of the sample 101 observed on site.

4.3. Plasticity and grain size analysis

Test rollers were modelled from the clayey samples in order to estimate the shrinkage. Two samples (101 and 107) were inappropriate for modelling a test roller due to their low plasticity and/or higher sand fraction. The results do not show the clear correlation between the plasticity range, grain size distribution and/or the shrinkage value (Table 5) as it is affected by several factors including the type of clay minerals, chemical composition, crystallinity, pH, impurities etc. rather than just the particle size or shape (GUGGENHEIM &

Table 5. List of clayey samples from the Staro Čiče-Gradišće site w	vith sampling depth, plasticity, Munsell (colour and shrinkage values (/ - not measured).

Sample number	Sampling depth (cm)	Plasticity	I	Munsell Color	Shrinkage (%)
99	100	moderate	10 YR 3/1	very dark grey	35
100	120	moderate	10 YR 3/1	very dark grey	32
101	115	no plasticity	5Y 4/2	olive grey	/
102	50	moderate	10 YR 4/2	dark greyish brown	33
103	60	low	10 YR 4/2	dark greyish brown	28
104	30	moderate	2.5Y 4/2	dark greyish brown	19
105	80	high	2.5Y 3/2	very dark greyish brown	49
106	140	moderate	2.5Y 3/1	very dark greyish brown	32
107	90	low	2.5Y 3/1	very dark greyish brown	/
108	50	high	2.5Y 5/3	light olive brown	28

MARTIN, 1995; SEMIZ, 2017; MORENO-MAROTO & ALONSO-AZCÁRATE, 2018).

According to the grain size distribution of the clayey samples collected in the vicinity of the Staro Čiče-Gradišće site, more than 90 vol. % of the particles in all of the samples are in a range between 4 and 113 µm. The majority of samples have more than 20 vol. % of sandy fraction (22 % - 42.50 vol. %) and less than 10 vol. % of clay. The remaining samples are characterized by their lower sand content (2 - 17.50 vol. %)and slightly greater amounts of clay (19 - 30 vol. %). All of the samples have the highest proportion of silt (52 - 72 vol. %) and can be therefore determined as sandy or clayey silt. However, the non-spherical shape of the clay particles combined with measurement limits, led to a different evaluation increasing the silt fraction (FEDOTOV et al., 2007; POLAKOWSKI et al., 2014; MAKO et al., 2017). Taking that into account, all of the samples may be assigned as sandy or silty clay as confirmed by the optical microscopy.

4.4. Geochemical analysis

The major, minor, and rare earth element (REE) contents were measured to identify the element concentration patterns among both the ceramics (27 samples) and clayey material (five samples, Suppl. 1). Major oxide concentrations revealed some variations among the clayey and the ceramic samples (Suppl. 1). The low CaO content in the clayey material collected near the Kurilovec-Belinščica site (< 1 %) distinguishes the samples from those collected near Staro Čiče-Gradišće which are characterized by higher CaO values (up to 7.6 %) (Fig. 6a, b). These clayey samples, related to the Staro Čiče-Gradišće, are further distinguished from others according to their higher MgO (around 3 %) content which may reflect dolomite in the samples. The concentrations of other major oxides are generally uniform among the clayey samples. The abundance of SiO₂ (~59.65 %) may be related to the high quartz, feldspar, and illite/muscovite content (Fig. 6a).

The ceramic sherds from Staro Čiče-Gradišće are discriminated from those found at the Kurilovec-Belinščica site by their higher CaO (~5.68 %) content. The abundance of CaO is commonly followed by the higher Sr, a typical isomorphic element in Ca-bearing minerals (GONZÁLEZ et al., 2018; GUTSUZ et al., 2017). The very high concentration of CaO and Loss on Ignition (LOI) in sample 277 (Suppl. 1) is the result of shell-tempered ceramics whereas the amount of SiO₂ in the ceramics is likely controlled by different types of igneous and metamorphic rocks added as a temper. Therefore, the lower SiO₂ is in general recorded in the samples without or with a lesser amount of lithoclast temper (samples 265, 277, 278, 280 and 285). The P₂O₅ content is below 5 % except for sample 14147. This probably reflects post-depositional

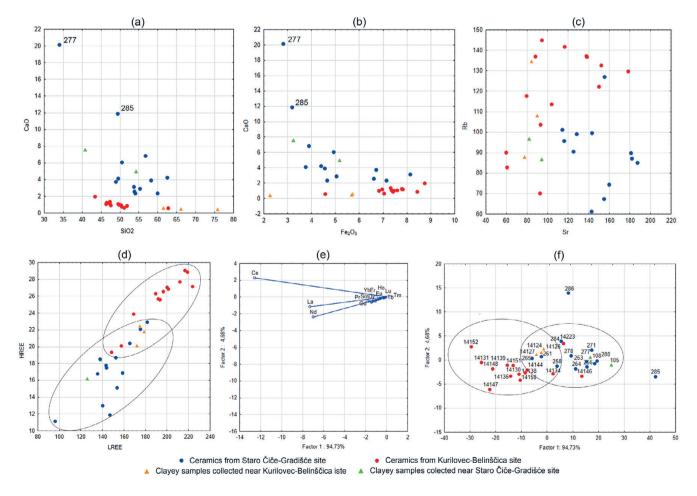


Figure 6. Bivariate plots of (a) SiO₂ vs CaO, (b) Fe₂O₃ vs CaO, (c) Sr vs Rb, (d) LREE (La, Ce, Pr, Nd, Sm, Eu) vs HREE (Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu), (e) projection of the variables on the factor-plane and (f) principal component analysis based on REE conducted on ceramics from the Staro Čiče-Gradišće and Kurilovec-Belinščica site and on clayey samples.

alterations (FREESTONe et al., 1994; from SANTOS RODRIGUES et al., 2015) although some authors relate the phosphorus level primarily to the use of pots for cooking (COSTA et al., 2011). The samples from Staro Čiče-Gradišće are slightly enriched in Sr if compared with ceramics from the Kurilovec-Belinščica site (Fig. 6c). Although the concentration of the major and minor elements is commonly affected by tempers that potters add to the raw clay, the rare earth elements tend to be allocated to the reactive surface of the clay minerals (CARLONI et al., 2021 and references therein) which makes them suitable for investigating the provenience of the clayey raw materials. Moreover, their concentration patterns do not significantly alter after the firing process (GUTSUZ et al., 2017). All of the samples have LREE > HREE. A diagram showing the total amount of light REE (La-Eu) and heavy REE (Gd-Lu) exposed two separate groups that generally correspond to different archaeological sites; the REE-poor sherds from the Staro Čiče-Gradišće site and REE-rich sherds from Kurilovec-Belinščica site (Fig. 6d). The clayey samples collected near Staro Čiče-Gradišće have similar REE composition to the ceramic sherds from the Staro Čiče-Gradišće site whereas those collected near Kurilovec-Belinščica site corresponds to the sherds from Kurilovec-Belinščica site. Principal component analysis based on REE concentrations shows samples' distribution mainly according to the Ce, La and Nd concentrations (Fig. 6e). A scatter plot of the component scores revealed a single cloud but with samples from Staro Ciče-Gradišće having higher factor 1 values if compared with the ceramics from Kurilovec-Belinščica. Two Neolithic samples (285 and 286) represent outliers (Fig. 6f).

5. DISCUSSION

5.1. Clayey raw material

Investigations of pottery practices have shown that the process of procuring clayey material is mainly reduced to the minimal expenditure of time and a minimal distance from the place of production (ARNOLD, 1985, 2005; GOSSELAIN & LIVINGSTONE SMITH, 2005). According to ethnographic examples, the clayey materials can be extracted using four main methods (surface, pit, gallery, and underwater extraction) depending on the characteristics of the surrounding area. Therefore, following the alluvial characteristics of the Turopolje region it is assumed that the raw clays used for pottery production was procured in a nearby environment including floodplains, fields, marshes, riverbeds, etc., mainly as surface or underwater extraction (GOSSELAIN & LIVINGSTONE SMITH, 2005). This is confirmed by the results of this study, which point to the local origin of the raw materials. Comparison of the geochemical patterns of ceramics with the clayey samples shows that the ceramics from the Kurilovec-Belinščica site are most similar to the clayey samples collected in the brickyard located 3 km from the site. Similarly, the ceramics from the Staro Čiče-Gradišće correspond well with the clayey samples collected up to 0.5 km from the site. The collected clays are flood deposits with similar mineralogical composition and are characterized by moderate or high plasticity, which initially makes them suitable for pottery making. Additionally, alluvial characteristics of the area suggest the collected samples are secondary clays transported from the parent rock (VELDE & MEUNIER, 2008).

Apart from the clayey samples, ceramic sherds are characterized by similar mineralogical composition observed by XRPD analysis. However, optical microscopy of thin sections indicated differences in the size and amount of crystalloclasts in the matrix resulting in five determined fabric groups: from inclusion-poor, very plastic clay to the inclusionrich, moderately plastic clay. The variability of these inclusions is not necessarily associated with different source locations but may indicate different depths of the raw clay exploitation. Namely, deposition of the larger mineral grains, mostly of quartz and feldspar, causes their enrichment with increasing depth. Inclusion-poor clays are more common in the surface sediments and are characterized by greater amounts of the swelling clay minerals which is confirmed by both the mineralogical and grain size analyses.

However, research has shown that particular communities from the past preferred one type of raw material over another. Some patterns, concerning the proportion of sandy and inclusion-poor clay, can be observed by comparing thinsections of pottery from the Staro Ciče-Gradišće but also those of the Bronze Age ceramics from Kurilovec-Belinščica site (KUDELIC & SIROVICA, 2022). The comparison shows that the most common type of clayey material during almost all the past periods is sandy clay with 15 - 30 % of very fine crystalloclast inclusions in the matrix. Inclusion-poor or fat clays (less than 10 % of crystalloclasts) were less common but very frequently used during the Bronze Age. Very sandy clay (more than 40 % of crystalloclasts) was sporadically present in the Bronze Age but more commonly used by potters during the Neolithic and the Late Antiquity periods. Therefore, based on the findings of this research, it is concluded that communities throughout all periods of the past exploited clayey materials from the vicinity of the settlement, as confirmed by the characteristics of the samples collected in the immediate environment.

5.2. Temper material

The study of raw materials used in the past is challenging because potters usually modify the original raw material in order to change its properties and adapt it to their needs. Therefore, during analysis, it is important to distinguish the original from the added or modified material. Pottery mixtures usually consist of two elements: raw clay as the basic element and temper that potters add to the clay (crushed rocks, minerals, ceramics or plant materials). The significance of temper material is manifested through its techno-functional role. They improve the viscosity of the clay paste, the strength of the vessel and the resistance to thermal stress (RICE, 1987; VELDE & DRUC, 1999) or directly affect the properties of the final product (ALBERO SANTACREU, 2014).

The results showed that potters used different temper materials the choice of which is mostly very characteristic for a particular period of the past, i.e. for particular communities. The most common temper material are lithoclasts: sub-angular to sub-rounded fragments of various sedimentary, igneous and metamorphic rocks. These are used by the communities from

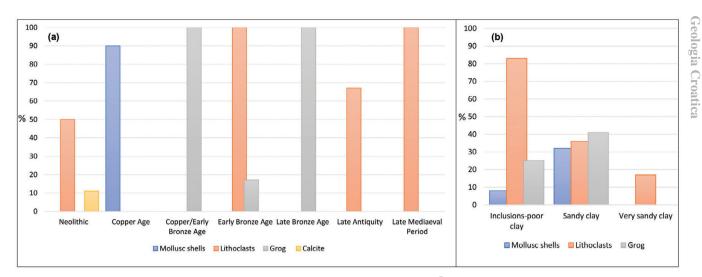


Figure 7. (a) the proportion of different types of tempering material in ceramics from the Staro Čiče-Gradišće site in relation to different periods of the past. (b) the proportion of different types of tempering material in ceramics from the Staro Čiče-Gradišće and Kurilovec-Belinščica site in relation to different types of the clayey raw material.

the Neolithic, Early Bronze Age, Late Antiquity and Late Mediaeval Periods (Fig. 7a). The variability of the lithoclasts and the sub-rounded shape indicate that these materials are sand and gravel transported from rivers and smaller watercourses, probably from riverbed gravel bars at the bank of the nearby river. According to the investigation of the provenance of pebbles from the Sava River (a nearby gravel pit located around 1 km from the Staro Ciče-Gradišće site), carbonates are dominant with a smaller proportion of igneous rocks (BARUDŽIJA et al., 2020). The pebble characteristics correspond to the rock temper present in ancient ceramics, particularly those from the Late Antiquity and Late Mediaeval periods. Locally available temper material can also be assumed for the Copper Age community using crushed mollusc shells that were probably exploited from the nearby rivers and watercourses. Such a choice of raw materials and such abundantly used temper have not been previously recorded in a wider area of research, representing the valuable evidence of specific technological praxis present among the community from the Copper Age. Late Copper/Early Bronze Age together with Late Bronze Age potters made exclusively grog-tempered pottery (KUDELIĆ et al., 2018; KUDELIĆ & SIROVICA, 2022; NERAL et al., 2023). However, four samples contain grog in addition to shell temper.

Although the local origin (of both clays and tempers) is confirmed for most of the ceramics, the sole exceptions are the Neolithic samples. Namely, a diverse geochemical pattern of two samples suggests a different source of clayey material. Additionally, the third sample is calcite tempered. Calcite is not of local origin considering the geological framework of the area (CGS, 2009), although its presence corresponds well with the ceramics from the Eastern Adriatic coast, i.e. karst environment (ŽIBRAT GAŠPARIČ, 2004; DOLENEC et al., 2012; SPATARO, 2002; KUDELIĆ et al., 2023). This strongly suggests either exploiting resources from more distant locations or that exchange networks were present among the Neolithic communities.

According to previous studies and considerations (KREITER, 2007; ALBERO SANTACREU, 2014), the choice

of pottery raw materials, especially temper, can be culturally conditioned, which partly rejects environmental and technofunctional reasons. This research has shown that the choice of raw materials was certainly conditioned by resource' availability in the immediate environment. However, despite the easily available and moderately plastic sandy clay, Bronze Age communities from Turopolje also commonly exploited specific sources of the inclusion-poor, highly plastic clay. This kind of clayey material has a higher proportion of the swelling clay minerals that cause significant volume change that can negatively affect the final product. In order to achieve optimal plasticity and reduce the overall volume change these clays are most commonly tempered with non-plastic materials (VELDE, 1992) which has also been supported by this research (Fig. 7b). However, according to the research results of pottery raw materials of Bronze Age communities from continental Croatia, preference is given to sandy clay (NERAL et al., 2023). Therefore, it can be assumed that the choice of inclusionpoor clay is of local significance.

6. CONCLUSION

This research presents the analyses of ancient ceramics from the multi-period archaeological sites in continental Croatia and potential clayey raw materials collected in the immediate environment and experimental clayey briquettes made from the collected clay. The study employed a multi-analytical approach to examine paste recipes, characterise raw materials, and explore their provenance, availability, and spatial distribution. Several key conclusions emerged from this investigation:

- Ancient potters preferred to use the moderately plastic, sandy clay, while highly plastic clay is frequently exploited only by the Bronze Age potters
- The potters used various tempering materials related to particular social groups and/or different periods of the past
- The most commonly used tempers, characteristic of the Neolithic, the Early Bronze Age, the Late Antiquity, and

the Late Mediaeval Period are sands and gravels exploited from the nearby rivers. Grog is characteristic only of the Bronze Age. Copper Age potters extensively used mollusc shells as a temper, which strongly indicates a specific technological praxis present among this community

- Most of the ceramics are of local origin, made from readily available raw materials present in the landscape composed of flood sediments of the nearby Sava River
- The only non-local raw materials were detected in the Neolithic samples, as evidenced by the use of non-local calcite temper and their different geochemical composition.

The study demonstrates that the investigated area possesses clay suitable for pottery production, thereby supporting the prevalent use of local sources. However, a more pronounced variety was recorded in the selection of tempering material, conditioned by the improvement of technological features but also by the regional pottery traditions specific to certain communities of the past.

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) K ₂ O (%) II	1.68 0.56	1.88 0.66	1.92 1.00	1.54 0.98	2.24 0.95	1.83 0.59	2.03 0.63	1.89 0.75	2.26 0.77	2.06 0.67	1.94 0.72	1.16 1.14	1.16 0.61	1.30 0.89	1.60 0.67	1.74 1.00	1.14 0.56	2.01 0.89	2.33 0.85	1.42 0.97	1.61 0.85	2.28 0.90	2.13 0.91	2.30 0.96	2.56 0.86	1.56 1.06	2.21 0.90	2.23 0.90	2.15 0.85	2.16 0.98	2.26 0.96	1.57 0.88	3.53 0.86	1.93 0.84
0 (0	7.60 0.62	5.01 0.81	0.48 0.88	0.44 0.74	0.60 0.76	3.87 0.59	4.21 0.65	6.82 0.76	2.56 0.69	2.31 0.42	6.02 0.72	3.10 0.38	20.13 0.24	3.69 0.14	4.09 0.20	2.34 0.56	11.84 0.59	2.86 0.14	0.87 0.44	1.96 0.35	1.07 0.46	1.16 0.45	0.84 0.56	0.95 0.45	1.25 0.46	0.62 0.53	1.34 0.49	1.03 0.50	1.14 0.45	0.72 0.29	0.96 0.54	1.19 0.79	0.86 0.34	1.56 0.35
%) M	3.23 2.88	5.16 3.37	5.68 1.12	2.26 0.68	5.71 1.29	4.56 1.55	4.41 1.50	3.89 1.31	6.61 1.63	4.66 1.08	4.93 1.66	8.15 0.68	2.83 0.60	6.72 1.17	3.76 0.99	7.15 0.88	3.20 0.68	5.05 0.57	8.43 1.38	8.75 1.30	7.60 1.27	7.82 1.10	7.42 1.40	7.45 1.19	7.79 1.28	7.05 0.78	7.32 1.06	7.36 1.29	6.96 1.53	5.15 0.80	6.83 0.94	4.81 0.67	8.12 1.60	7.46 0.74
(0)	11.77 3	13.00 5	15.04 5	11.86 2	16.22 5	17.32 4	15.39 4	14.21 3	17.96 6	18.02 4	17.33 4	16.47 8	13.24 2	19.57 6	22.11 3	18.20	11.21 3	17.82 5	22.23 8	23.51 8	22.47 7	21.73 7	20.43 7	22.32	21.14 7	20.78 7	19.98 7	20.29 7	21.66 6	21.06 5	19.78 6	18.70 4	22.26 8	21.96 7
	SČG 40.78	SČG 54.18	K-B 66.10	K-B 75.78	K-B 61.39	SČG 58.22	SČG 62.55	SČG 56.83	SČG 53.79	SČG 59.95	SČG 50.54	SČG 53.75	SČG 34.06	SČG 48.96	SČG 49.52	SČG 54.12	SČG 49.43	SČG 55.25	K-B 47.58	K-B 43.41	K-B 46.36	K-B 46.65	K-B 51.88	K-B 49.77	K-B 46.48	K-B 51.13	K-B 47.40	K-B 49.57	K-B 47.37	K-B 50.57	K-B 50.31	K-B 58.69	K-B 50.26	K-B 46.19
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imber sam	105 cla	108 cla	14124 cla	14126 cla	14127 cla	253 cerai	256 cerai	258 cerai	261 cerai	264 cerai	265 cerai	271 cerai	277 cerai	278 cerai	280 cerai	284 cerai	285 cerai	286 cerai	14130 cerai	14131 cerai	14134 cerai	14135 cerai	14138 cerai	14139 cerai	14144 cerai	14146 cerai	14147 cerai	14148 cerai	14150 cerai	14151 cerai	14152 cerai	14154 cerai	14160 cerai	14161 cerai

atica	Su	ppl	em	ent	1.	– C	on	tinu	led	
Geologia Cro	Ta (ppm)	0.80	0.90	1.20	1.30	1.30	0.80	0.90	1.10	

Sample number Sample type	r Sample type	Site	Ni (ppm)	Sc (ppm)	Ba (ppm)	Be (ppm)	Co (ppm)	Cs (ppm)	Ga (ppm)	Hf (ppm)	(mqq) qN	Rb (ppm)	Sn (ppm)	Sr (ppm)	Ta (ppm)
105	clay	SČG	44.00	11.00	426.00	$\overline{\nabla}$	15.70	6.70	19.00	4.20	10.10	86.80	3.00	94.20	0.80
108	clay	SČG	38.00	12.00	340.00	1.00	10.30	5.60	14.70	5.10	10.80	96.80	3.00	81.80	06.0
14124	clay	K-B	37.00	14.00	445.00	7	13.60	8.30	20.70	8.40	17.70	108.00	4.00	89.50	1.20
14126	clay	K-B	27.00	10.00	297.00	2.00	8.20	6.00	13.80	10.40	18.70	87.90	3.00	77.50	1.30
14127	clay	K-B	45.00	15.00	289.00	<1	10.00	6.60	15.00	7.00	16.80	134.50	4.00	84.40	1.30
253	ceramics	SČG	50.00	16.00	446.00	2.00	9.70	7.10	19.10	3.50	10.10	98.90	4.00	128.50	0.80
256	ceramics	SČG	37.00	14.00	449.00	4.00	10.00	6.60	17.00	3.80	11.00	95.60	4.00	116.40	0.90
258	ceramics	SČG	33.00	13.00	768.00	6.00	10.50	5.00	14.30	5.40	12.30	86.90	3.00	182.20	1.10
261	ceramics	SČG	74.00	17.00	738.00	7.00	14.30	8.70	19.50	4.70	12.60	126.90	5.00	155.60	0.80
264	ceramics	SČG	59.00	16.00	789.00	7	10.90	7.60	18.90	3.80	11.50	99.40	4.00	143.10	0.90
265	ceramics	SČG	73.00	17.00	963.00	1.00	12.60	6.20	18.90	4.80	12.40	89.70	4.00	181.40	1.00
271	ceramics	SČG	36.00	18.00	557.00	2.00	10.00	5.00	18.60	6.30	14.90	61.10	3.00	142.90	1.20
277	ceramics	SČG	40.00	13.00	719.00	7	5.40	5.20	15.00	3.40	11.00	85.00	3.00	187.40	0.80
278	ceramics	SČG	88.00	18.00	911.00	3.00	12.90	8.10	22.90	3.80	15.00	74.30	5.00	160.20	1.10
280	ceramics	SČG	61.00	19.00	647.00	5.00	11.20	8.40	25.50	3.90	11.90	101.00	5.00	114.50	0.80
284	ceramics	SČG	43.00	17.00	778.00	3.00	5.40	7.70	20.50	7.40	18.20	90.30	4.00	125.50	1.40
285	ceramics	SČG	32.00	10.00	935.00	<1	7.60	3.30	10.90	3.40	9.00	49.90	3.00	211.40	0.80
286	ceramics	SČG	23.00	18.00	889.00	3.00	4.70	4.30	19.10	5.90	16.30	67.30	6.00	155.00	1.20
14130	ceramics	K-B	00.66	21.00	948.00	<1	21.10	11.00	27.70	4.00	15.20	144.80	4.00	94.80	0.90
14131	ceramics	K-B	63.00	21.00	619.00	2.00	21.70	5.20	24.30	4.90	17.10	70.10	5.00	92.80	1.40
14134	ceramics	K-B	103.00	21.00	795.00	5.00	15.80	10.10	27.20	4.80	15.20	117.50	5.00	79.90	1.10
14135	ceramics	K-B	61.00	21.00	1374.00	6.00	16.40	8.10	26.00	5.10	16.50	122.10	5.00	150.30	1.30
14138	ceramics	K-B	87.00	20.00	840.00	4.00	15.60	9.80	24.40	5.40	16.70	136.80	4.00	88.20	1.10
14139	ceramics	K-B	64.00	21.00	773.00	6.00	15.60	7.70	25.60	5.20	16.10	113.50	5.00	103.90	1.50
14144	ceramics	K-B	74.00	21.00	1065.00	3.00	15.10	10.00	25.40	4.50	14.90	136.60	4.00	138.30	1.10
14146	ceramics	K-B	82.00	19.00	557.00	5.00	9.80	8.90	23.20	6.40	19.20	89.90	5.00	59.80	1.40
14147	ceramics	K-B	70.00	19.00	1724.00	3.00	15.50	8.80	24.10	5.10	16.90	129.50	5.00	178.60	1.20
14148	ceramics	K-B	74.00	20.00	1304.00	2.00	18.90	9.40	25.80	5.50	16.30	137.00	5.00	138.00	1.20
14150	ceramics	K-B	97.00	21.00	1339.00	5.00	14.10	10.70	26.50	4.50	14.60	141.60	4.00	116.50	1.10
14151	ceramics	K-B	63.00	19.00	939.00	6.00	10.50	7.80	24.30	6.10	17.30	103.60	4.00	93.30	1.10
14152	ceramics	K-B	73.00	20.00	1084.00	1.00	17.80	9.50	23.90	6.40	19.00	132.50	4.00	152.20	1.10
14154	ceramics	K-B	57.00	15.00	468.00	3.00	11.40	5.60	19.10	6.90	15.10	79.80	4.00	77.60	1.20
14160	ceramics	K-B	116.00	21.00	704.00	5.00	16.90	11.80	25.70	4.40	15.60	132.70	5.00	61.20	1.20
14161	ceramics	K-B	66.00	18.00	409.00	2.00	11.50	6.50	22.60	4.80	14.70	85.00	4.00	50.80	1.00
14223	ceramics	K-B	69.00	15.00	488.00	2.00	13.70	6.10	19.00	5.70	14.30	82.60	3.00	60.70	1.00

Sample number Sample type	er Sample type	Site	Th (ppm)	(mdd) U	V (ppm)	(mqq) W	Zr (ppm)	Y (ppm)	La (ppm)	Ce (ppm)	Pr (ppm)	(mdd) pN	Sm (ppm)	Eu (ppm)	Gd (ppm)
105	clay	SČG	10.50	3.90	94.00	1.30	146.10	25.20	30.00	57.90	6.77	25.30	4.85	0.96	4.60
108	clay	SČG	11.70	2.70	92.00	2.50	185.30	29.00	32.60	63.20	7.37	28.00	5.67	1.15	5.17
14124	clay	K-B	12.50	3.80	121.00	2.20	338.20	32.90	40.2	80.80	9.52	36.00	6.73	1.44	6.42
14126	clay	K-B	11.70	3.60	87.00	2.70	400.60	30.50	41.90	79.80	8.86	34.00	6.07	1.18	5.36
14127	clay	K-B	13.40	3.60	128.00	2.90	250.40	31.30	42.10	81.60	9.67	36.60	7.01	1.37	6.37
253	ceramics	SČG	13.60	3.40	92.00	1.60	114.00	27.20	34.30	65.70	7.73	28.90	5.87	1.15	5.32
256	ceramics	SČG	13.60	3.50	95.00	1.60	143.10	25.60	34.10	65.10	7.79	30.00	5.69	1.14	5.15
258	ceramics	sčg	12.70	2.90	78.00	1.80	202.50	31.90	39.20	74.10	9.06	35.00	6.77	1.38	5.99
261	ceramics	SČG	16.30	3.00	107.00	2.00	173.20	32.80	41.10	80.00	9.67	36.20	7.13	1.41	6.52
264	ceramics	SČG	13.50	3.50	152.00	2.20	134.70	28.00	36.60	68.10	8.38	31.90	6.32	1.26	5.51
265	ceramics	SČG	15.80	2.70	107.00	1.90	185.20	34.80	42.90	82.50	9.90	37.80	7.20	1.54	7.00
271	ceramics	SČG	12.50	2.80	140.00	3.20	253.40	27.40	31.30	66.00	7.17	27.10	5.41	1.04	4.83
277	ceramics	SČG	14.40	2.70	89.00	1.90	142.20	18.10	34.50	64.80	7.58	27.50	4.97	1.03	3.99
278	ceramics	SČG	12.80	4.70	145.00	2.10	142.10	22.60	37.90	70.70	8.16	30.50	5.45	1.02	4.40
280	ceramics	SČG	15.50	3.60	133.00	2.00	142.10	25.50	33.10	62.60	7.15	26.70	5.06	1.01	4.70
284	ceramics	SČG	21.10	3.70	130.00	4.10	285.10	25.70	36.80	76.10	8.20	31.10	5.61	1.02	4.98
285	ceramics	SČG	8.40	2.10	55.00	1.80	137.30	17.80	23.30	43.30	5.20	19.70	3.88	0.73	3.24
286	ceramics	SČG	12.30	3.70	117.00	2.40	214.40	16.90	32.60	79.90	6.62	22.90	4.17	0.78	3.39
14130	ceramics	K-B	15.80	4.30	163.00	2.90	147.10	35.40	45.80	84.40	10.74	42.50	8.33	1.68	7.82
14131	ceramics	K-B	17.70	3.40	171.00	2.70	179.40	39.10	52.00	97.00	12.09	46.70	9.49	1.91	8.77
14134	ceramics	K-B	15.20	4.30	166.00	2.20	178.30	32.30	38.60	74.60	9.69	37.20	7.42	1.55	7.20
14135	ceramics	K-B	16.00	4.90	144.00	2.50	186.00	36.70	48.10	86.50	11.38	43.70	8.64	1.82	8.08
14138	ceramics	K-B	15.50	3.80	161.00	1.60	198.00	37.30	45.00	82.80	11.00	40.70	8.44	1.71	8.00
14139	ceramics	K-B	16.20	4.50	150.00	2.90	191.40	38.60	46.50	89.30	11.54	43.50	8.66	1.70	8.18
14144	ceramics	K-B	14.60	3.60	156.00	2.10	155.30	37.50	45.10	82.30	10.58	39.70	7.90	1.68	7.72
14146	ceramics	K-B	17.60	4.60	165.00	2.60	238.20	28.30	34.20	65.60	8.34	33.00	6.25	1.30	5.70
14147	ceramics	K-B	15.10	4.30	149.00	2.40	187.40	42.40	54.00	90.70	12.61	48.20	9.24	2.00	9.21
14148	ceramics	K-B	15.10	4.30	143.00	2.10	189.60	41.50	50.60	92.80	12.00	45.70	9.09	1.91	8.59
14150	ceramics	K-B	14.90	4.30	156.00	2.40	157.10	37.90	46.60	83.30	10.81	43.00	8.16	1.61	7.76
14151	ceramics	K-B	15.80	3.70	136.00	2.50	215.50	36.80	47.00	87.10	10.89	41.60	8.35	1.57	7.75
14152	ceramics	K-B	16.10	5.10	139.00	2.40	224.30	41.60	54.00	101.90	11.83	45.90	8.44	1.74	8.32
14154	ceramics	K-B	14.00	10.30	130.00	2.10	240.80	34.10	36.90	71.30	8.83	34.60	7.08	1.43	6.61
14160	ceramics	K-B	17.70	4.40	145.00	2.40	159.80	29.90	34.10	68.60	8.66	33.50	7.16	1.47	6.43
14161	ceramics	K-B	15.70	9.20	144.00	2.20	168.10	35.40	43.40	76.20	10.20	38.80	8.23	1.59	7.22
14223	ceramics	K-B	12.60	5.20	122.00	1.90	203.50	29.50	34.60	75.50	8.28	31.60	6.35	1.28	5.86

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Supplement 1. – Continued

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As (ppm)	2.40	9.40	11.00	1.80	10.50	3.80	3.60	4.00	12.10	6.60	12.80	11.80	7.40	10.30	10.00	40.00	9.80	20.70	13.60	11.00	11.70	8.30	11.60	00.6	16.00	15.90	10.00	10.70	10.90	11.70	11.90	8.20	9.60	8.20	10.30
Ni (ppm)	30.30	29.30	23.90	11.60	31.00	33.80	32.40	30.30	61.70	35.00	48.00	29.50	27.10	63.50	31.60	37.00	29.30	20.30	79.00	42.10	75.60	44.30	60.30	41.10	62.40	56.60	56.80	59.20	73.60	38.60	50.10	44.00	102.80	59.70	44.20
(mqq) nZ	72.00	61.00	60.00	30.00	71.00	116.00	94.00	133.00	175.00	110.00	155.00	61.00	75.00	159.00	112.00	97.00	95.00	76.00	131.00	88.00	121.00	125.00	110.00	87.00	135.00	60.00	131.00	128.00	147.00	83.00	103.00	68.00	150.00	116.00	67.00
(mqq) dq	21.10	18.80	18.40	13.30	27.10	16.50	16.70	15.30	21.70	17.70	19.00	19.10	16.90	23.90	18.90	20.20	12.80	25.00	34.80	36.00	28.60	26.40	26.00	27.90	33.00	25.20	28.70	30.30	29.40	27.90	25.80	29.20	39.90	34.80	25.10
Cu (ppm)	32.00	18.80	17.10	9.80	19.00	23.70	20.50	24.30	35.40	23.80	38.30	25.90	21.10	22.70	26.00	13.60	18.80	12.00	56.60	32.60	40.00	26.60	29.20	24.40	38.90	28.10	41.80	36.60	40.00	39.60	31.20	24.10	39.30	36.90	21.20
(mdd) oM	0.60	0.70	0.50	0.10	0.50	0.30	0.30	0.40	1.10	0.30	0.40	0.60	0.20	0.50	0.10	0.20	0.40	1.00	0.40	0.30	0.40	0.40	0.40	0.30	0.60	0.30	0.40	0.40	0.40	0.20	0.40	0.20	1.20	0.20	0.20
(mdd) nJ	0.37	0.40	0.51	0.52	0.49	0.37	0.35	0.45	0.45	0.39	0.47	0.43	0.26	0.33	0.35	0.37	0.23	0.25	0.53	0.59	0.51	0.56	0.58	0.59	0.58	0.44	0.54	0.61	0.55	0.59	0.58	0.51	0.53	0.53	0.47
Yb (ppm)	2.38	2.61	3.38	3.20	3.15	2.55	2.56	2.89	3.06	2.66	3.12	2.88	1.87	2.26	2.60	2.54	1.58	1.77	3.58	4.16	3.34	3.89	3.63	3.91	3.56	2.82	4.14	4.00	3.55	3.79	3.83	3.20	3.47	3.59	3.06
Tm (ppm)	0.36	0.41	0.54	0.47	0.49	0.39	0.34	0.45	0.47	0.40	0.46	0.44	0.24	0.35	0.38	0.36	0.23	0.26	0.56	0.62	0.50	0.60	0.58	0.61	0.57	0.44	0.62	0.62	0.56	0.59	0.56	0.49	0.51	0.55	0.44
Er (ppm)	2.53	2.94	3.49	3.39	3.27	2.69	2.76	3.16	3.36	2.89	3.45	3.01	1.83	2.40	2.74	2.67	1.74	1.89	3.77	4.29	3.58	4.07	4.04	3.83	3.80	2.89	4.07	3.98	3.77	4.14	4.17	3.55	3.35	3.69	3.14
(mdd) oH	0.87	1.03	1.18	1.08	1.20	0.92	0.92	1.16	1.16	0.94	1.19	0.99	0.66	0.76	0.83	0.87	0.56	0.61	1.25	1.48	1.22	1.41	1.32	1.46	1.36	1.03	1.50	1.45	1.33	1.48	1.47	1.22	1.23	1.29	1.08
Dy (ppm)	4.35	5.08	5.96	5.22	5.81	4.71	4.59	5.35	6.04	5.07	6.21	5.15	3.54	3.93	4.44	4.36	3.07	3.18	6.84	7.63	6.38	7.18	6.89	7.03	6.62	5.13	7.56	7.12	6.84	6.96	6.98	5.91	6.08	6.40	5.07
Tb (ppm)	0.72	0.83	1.02	06.0	1.01	0.80	0.77	0.91	0.98	0.83	1.02	0.77	0.60	0.65	0.71	0.72	0.47	0.51	1.22	1.32	1.13	1.25	1.24	1.24	1.18	0.85	1.39	1.34	1.22	1.23	1.23	1.01	1.06	1.17	0.94
Site	SČG	SČG	K-B	K-B	K-B	SČG	K-B																												
Sample type	clay	clay	clay	clay	clay	ceramics																													
Sample number Sample type	105	108	14124	14126	14127	253	256	258	261	264	265	271	277	278	280	284	285	286	14130	14131	14134	14135	14138	14139	14144	14146	14147	14148	14150	14151	14152	14154	14160	14161	14223

Supplement 1. – Continued

105	clay	SČG	0.50	0.30	0.30	<0.1	2.10	0.13	0.10	5.90
108	clay	SČG	0.20	0.30	0.20	<0.1	1.80	0.10	0.10	0.80
14124	clay	K-B	<0,1	0.40	0.30	<0,1	2.20	0.08	0.20	<0,5
14126	clay	K-B	<0,1	0.20	0.20	<0,1	2.30	0.05	0.20	<0,5
14127	clay	K-B	0.30	0.30	0.30	<0,1	1.80	0.08	0.20	0.80
253	ceramics	SČG	0.20	0.10	0.30	<0.1	0.80	<0.01	0.30	<0.5
256	ceramics	SČG	0.20	0.20	0.10	<0.1	<0.5	0.02	0.30	<0.5
258	ceramics	SČG	0.50	0.20	0.10	<0.1	1.00	0.02	0.20	<0.5
261	ceramics	SČG	0.80	0.20	0.30	<0.1	1.00	0.04	0.20	<0.5
264	ceramics	SČG	0.30	0.20	0.20	<0.1	0.80	0.02	0.30	<0.5
265	ceramics	SČG	09.0	0.20	0.20	<0.1	3.00	0.03	0.20	<0.5
271	ceramics	SČG	0.30	0.40	0.20	<0.1	1.10	0.02	0.20	<0.5
277	ceramics	SČG	0.50	<0.1	0.20	<0.1	1.00	0.03	0.20	<0.5
278	ceramics	SČG	0.50	<0.1	0.30	<0.1	1.00	0.03	0.30	<0.5
280	ceramics	SČG	0.30	<0.1	0.30	<0.1	1.60	0.02	0.30	<0.5
284	ceramics	SČG	0.30	0.10	0.40	<0.1	0.70	0.14	0.20	<0.5
285	ceramics	SČG	0.80	0.20	0.20	<0.1	2.20	0.05	0.20	<0.5
286	ceramics	SČG	0.40	0.30	<0.1	<0.1	2.30	0.04	0.20	<0.5
14130	ceramics	K-B	0.50	0.20	0.50	0.10	2.30	0.02	0.30	0.60
14131	ceramics	K-B	0.60	0.40	<0,1	0.10	1.80	0.02	0.30	<0,5
14134	ceramics	K-B	0.30	0.20	0.40	0.10	1.00	0.01	0.30	0.50
14135	ceramics	K-B	0.50	0.30	<0,1	<0,1	1.20	0.01	0.40	<0,5
14138	ceramics	K-B	0.40	0.20	0.40	<0,1	2.80	0.02	0.30	0.80
14139	ceramics	K-B	0.30	0.30	0.20	0.10	1.70	0.03	0.40	0.60
14144	ceramics	K-B	0.70	0.20	0.40	0.10	1.10	0.02	0.30	0.70
14146	ceramics	K-B	0.30	0.20	0.30	0.10	2.30	0.01	0.30	<0,5
14147	ceramics	K-B	0.80	0.20	0.30	<0,1	1.40	0.02	0.30	0.50
14148	ceramics	K-B	0.70	0.20	0.30	<0,1	<0,5	0.02	0.30	0.70
14150	ceramics	K-B	0.60	0.10	0.40	0.10	2.00	0.02	0.30	0.60
14151	ceramics	K-B	0.40	0.20	<0,1	0.10	1.20	0.03	0.20	<0,5
14152	ceramics	K-B	0.50	0.20	0.20	<0,1	0.90	0.02	0.40	<0,5
14154	ceramics	K-B	0.30	0.20	0.30	<0,1	2.60	0.03	0.30	0.50
14160	ceramics	K-B	0.80	0.20	0.40	<0,1	2.80	<0,01	0.50	<0,5
14161	ceramics	K-B	0.40	0.50	<0,1	<0,1	1.40	0.02	0.20	0.60

Supplement 1. – Continued