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Faculty of Mining, Geology and Petroleum Engineering

Natali Neral

**PROVENIENCE AND PROPERTIES OF
THE RAW MATERIALS IN THE CONTEXT
OF POTTERY TECHNOLOGY FROM
DIFFERENT PERIODS OF THE PAST IN
CROATIA**

DOCTORAL DISSERTATION

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Supervisors:

Associate Professor Ana Maričić, PhD

Andreja Kudelić, Senior Research Associate, PhD

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Sveučilište u Zagrebu

Rudarsko-geološko-naftni fakultet

Natali Neral

**PODRIJETLO I SVOJSTVA SIROVINA U
KONTEKSTU TEHNOLOGIJE
PROIZVODNJE LONČARIJE IZ
RAZLIČITIH RAZDOBLJA PROŠLOSTI NA
PODRUČJU HRVATSKE**

DOKTORSKI RAD

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ABSTRACT

This doctoral research represents the first comprehensive and systematic investigation of pottery raw materials and production technologies across various prehistoric and historic periods (from the Neolithic to the Late Middle Ages) and different regions of Croatia (Eastern and Central Croatia, and Istria). The primary objective of the study is to identify the types, mineralogical, petrographic, physical and mechanical properties, and provenience of pottery raw materials used in the past. In addition, the research explores technological process of pottery production to provide a better understanding of ancient manufacturing practices. A multidisciplinary analytical approach was employed, integrating mineralogical, petrographic, geochemical, sedimentological, and mechanical methods. The clayey raw materials identified in the ceramics can be broadly categorized into two main groups: sandy clay and inclusion-poor clay (high-plasticity clay). Sandy clay, characterized by moderate plasticity and shrinkage, was the most commonly used raw material throughout all periods. The use of inclusion-poor clay was less common. This high-plasticity material, rich in clay minerals, shows greater shrinkage and is more prone to deformation during drying and firing due to intense water loss. Despite these challenges, it was particularly used by Bronze Age communities in Central Croatia. Ceramics made from inclusion-poor clays exhibit significantly higher point load strength than those from sandy clays. This likely suggests that sandy clays were widely used for their availability and workability, while inclusion-poor clays reflect a deliberate choice for better mechanical properties. Furthermore, nearly all analysed pottery samples contain added tempering materials (calcite, graphite, grog, sand, chaff, shells, lithoclasts). The use of tempers was primarily motivated by the need to enhance the physical and mechanical properties of the clay body and the final product, including improved workability, greater thermal conductivity, and increased resistance to thermal shock. Nevertheless, in certain contexts, the selection of specific temper types may also have been influenced by traditional practices. The analysis reveals significant variability in temper types across different periods and geographic regions. In Continental Croatia, a greater diversity of tempering materials was identified compared to the geologically distinct region of Istria, indicating that environmental factors played a crucial role in shaping technological decisions during pottery production. Most of the analysed ceramics were produced using locally available raw materials. Pottery made from non-local sources is rare, yet it provides valuable evidence for the potential influence of additional factors, including trade connections, migration, or other forms of socio-economic interaction.

PROŠIRENI SAŽETAK

Uvod

Doktorsko istraživanje predstavlja prvo sveobuhvatno i sustavno istraživanje lončarskih sirovina i tehnoloških postupaka proizvodnje lončarije kroz različita prapovijesna i povijesna razdoblja na području Hrvatske. Analiza lončarije iz perspektive znanosti o materijalima, koja podrazumijeva analizu sirovina (gline i primjesa) te rekonstrukciju tehnoloških procesa, omogućuje prikupljanje širokog spektra znanstveno utemeljenih podataka koji pridonose razumijevanju tehnoloških postupaka proizvodnje lončarije kroz prošlost. Takva su istraživanja multidisciplinarna te zahtijevaju integraciju različitih metodologija i suradnju između različitih znanstvenih područja i polja, ponajprije arheologije, geologije i geološkog inženjerstva, kako bi se postiglo sveobuhvatnije razumijevanje davnih zajednica i njihovih tehnologija. U tom multidisciplinarnom okviru, poseban naglasak u ovom doktorskom istraživanju stavljen je na primjenu geoloških i inženjersko-geoloških metoda u analizi arheoloških i geoloških materijala, s ciljem određivanja vrste, svojstava i podrijetla sirovina korištenih u lončarskoj proizvodnji, kao i razumijevanja pripadajućih tehnoloških procesa.

Ciljevi i hipoteze

Ciljevi ovog rada su: (1) utvrditi vrstu, podrijetlo i pojedina fizičko-mehanička svojstva te dostupnost pretpostavljenih sirovina koje su na području Hrvatske korištene za proizvodnju lončarije kroz različita razdoblja prošlosti, (2) provesti komparativnu analizu arheološke keramike i lončarskih sirovina iz okoliša, (3) utvrditi karakteristike tehnoloških postupaka (način pripreme lončarske smjese i tehnike pečenja) proizvodnje lončarije za svako istraživano razdoblje prošlosti te (4) istražiti promjenjivost u izborima sirovina i tehnologiji proizvodnje lončarije kroz različita razdoblja prošlosti. Navedeni ciljevi istraživanja temeljni su na hipotezama: (1) glinovita sirovina za izradu lončarije dobavljana je u neposrednoj blizini naselja u kojem se odvijala proizvodnja lončarije, (2) izbor lončarskih sirovina uvjetovan je njezinom dostupnošću i geološkom građom područja te (3) tehnološki postupci proizvodnje lončarije (način pripreme lončarske smjese i tehnike pečenja) nisu nužno uvjetovani optimizacijom tehnoloških rješenja.

Znanstveni doprinos

Istraživanje lončarskih sirovina i tehnoloških postupaka proizvodnje omogućit će detaljnije razumijevanje promjena u izboru materijala i tehnologiji izrade keramike kroz različite povijesne periode na području Hrvatske. Dobiveni rezultati pružit će uvid u drevne proizvodne procese te dinamiku korištenja prirodnih resursa unutar šireg prostorno-vremenskog okvira, uključujući različite regije i kulturne skupine iz različitih razdoblja prošlosti.

Materijali i metode

Doktorski rad usmjeren je na istraživanje lončarskih sirovina i tehnologije proizvodnje lončarije kroz različita razdoblja prošlosti na području Republike Hrvatske. Predmet istraživanja je arheološka keramika (lončarija) s višeperiodnih arheoloških nalazišta (od neolitika do kasnog srednjeg vijeka) i lončarske sirovine (glinoviti materijal i primjese) prikupljene terenskim istraživanjima u blizini arheoloških nalazišta. Istraživanje obuhvaća tri studije slučaja. Svaka studija slučaja odnosi se na geografski i geološki različito područje, dok su u arheološkom kontekstu ta područja kroz prošlost bila izložena različitim društveno-gospodarskim i kulturnim utjecajima. Prva studija slučaja usmjerena je na područje Baranje odnosno arheološko nalazište Jagodnjak-Krčevine (Istočna Hrvatska). Druga studija slučaja primarno je usmjerena na područje Turopolja i nalazište Staro Čiče-Gradišće (Središnja Hrvatska) uz nadopunu podacima s obližnjeg nalazišta Kurilovec-Belinščica. Treća studija slučaja obuhvaća sjeverni dio istočnojadranske obale, s posebnim naglaskom na područje Istre. Kako bi se uspostavio reprezentativan arheološki uzorak koji obuhvaća različita razdoblja prošlosti, u okviru ove studije odabrana su tri nalazišta međusobno udaljena najviše 3 km: Stari Rakalj, Šiljar i Sveti Teodor.

Metodologija istraživanja podrazumijeva primjenu različitih analitičkih metoda koje omogućuju rekonstrukciju tehnoloških postupaka proizvodnje lončarije te uvid u vrste i podrijetlo sirovina korištenih u prošlosti. Terensko uzorkovanje potencijalnih glinovitih lončarskih sirovina provedeno je u radijusu do 10 km od arheoloških nalazišta, pri čemu je izbor lokacija temeljen na prethodnim istraživanjima i pretpostavljenoj udaljenosti koju su lončari prelazili u svrhu pribavljanja sirovine (Arnold, 2000; Rice 1987: 115–116). Prikupljeni glinoviti uzorci oblikovani su u testne pločice i pečeni u laboratorijskoj peći. Uzorci arheološke keramike i testne pločice keramike analizirane se metodom optičke mikroskopije tankih izbrusaka koristeći polarizacijski mikroskop. Metoda omogućuje prikupljanje podataka o mineraloško-petrografskom sastavu

uzoraka, načinu pripreme lončarske smjese (lončarskoj recepturi) te procjeni temperature pečenja. Uzorci keramike su zasebno za svako arheološko nalazište izdvojeni u grupe i podgrupe struktura keramike na temelju razlika u mineralnom sastavu i strukturi glinovitog matriksa koje posljedično ukazuju na različite vrste glinovitih sirovina odnosno primjesa (PCRG, 2010; Quinn, 2013).

Usporedba mineraloškog sastava i strukture matriksa uzoraka keramike i testnih pločica omogućila je približno određivanje glinovitih sirovina korištenih za izradu lončarije. Nadalje, određivanje mineraloškog sastava uzoraka keramike i gline provedeno je putem rendgenske difrakcije na prahu. Dodatno, na glinovitim je uzorcima izdvojena i analizirana frakcija $<2\text{ }\mu\text{m}$ s ciljem utvrđivanja pojedinih minerala glina prema smjernicama opisanima u Moore i Reynolds (1997). Na određenim uzorcima gline prikupljene u okolišu utvrđene su Atterbergove granice plastičnosti te preciznije definiranje vrste sedimenta metodom laserske granulometrije. Radi preciznijeg uvida u podrijetlo glinovitog materijala koji je korišten u prošlosti, na uzorcima keramike i gline iz okoliša provedene su i geokemijske metode uključujući masenu i emisijsku spektrometriju induktivno spregnute plazme. Geokemijski podaci statistički su obrađeni koristeći bivarijatne i multivarijatne statističke metode. Naposljetku, s ciljem uvida u varijabilnost tehnološkog postupka i mehanička svojstva keramike izrađena od različitih vrsta gline i primjesa, napravljeni su eksperimentalni uzorci keramike te je na njima provedeno određivanje indeksa čvrstoće pri opterećenju u točki. Dobiveni rezultati podvrgnuti su statističkoj analizi s ciljem procjene varijacija u vrijednostima čvrstoće unutar analiziranih skupova uzoraka.

Ključni rezultati i zaključci

Analiza lončarije iz različitih geografskih regija te prapovijesnih i povijesnih razdoblja na području Hrvatske omogućila je identifikaciju raznolikih vrsta sirovina koje su se koristile u lončarskoj proizvodnji. Na području kontinentalne Hrvatske utvrđeno je da su lončari najčešće koristili pjeskovite gline kao osnovnu sirovinu. Takve gline talože se u fluvijalnim okolišima visoke energije, što pogoduje sedimentaciji grubljih čestica u kombinaciji s mineralima glina. Pjeskovite gline posjeduju umjerenu plastičnost i skupljanje, svojstva koja ih čine pogodnima za lončarsku proizvodnju. S obzirom na njihovu dostupnost te povoljne karakteristike, pjeskovite gline bile su dominantna sirovina u proizvodnji lončarije kroz prapovijesna i povijesna razdoblja.

Uz pjeskovite gline, zabilježena je i uporaba glina visoke plastičnosti (masne gline), osobito na području središnje Hrvatske. Ove gline talože se u fluvijalnim okolišima niske energije

ili se formiraju kroz dugotrajne procese transporta i sortiranja, što dovodi do veće koncentracije minerala glina. Za takvu vrstu glinovitog materijala karakteristična je visoka plastičnost i znatno veće skupljanje, što se izravno povezuje s prisutnošću većeg udjela minerala glina. Tijekom sušenja i pečenja, gline visoke plastičnosti podložnije su deformacijama zbog intenzivnog gubitka vode, za razliku od pjeskovitih glina s nižim udjelom minerala glina. Ipak, unatoč tehnološkim izazovima, takve gline korištene su u lončarskoj proizvodnji, osobito unutar brončanodobnih zajednica središnje Hrvatske (Kudelić *et al.*, 2018; Neral *et al.*, 2024). Međutim, eksperimentalno istraživanje provedeno u okviru doktorskog istraživanja (Neral *et al.*, 2025) pokazalo je kako keramika izrađena od glina visoke plastičnosti ima značajno veću čvrstoću pri opterećenju u točki u odnosu na onu izrađenu od pjeskovite gline, što se pripisuje boljoj kompaktnosti čestica tijekom obrade, sušenja i pečenja. Ovi rezultati sugeriraju da je upotreba pjeskovitih glina prvenstveno bila rezultat njihove široke dostupnosti i lake obradivosti, dok se uporaba također lako dostupnih glina visoke plastičnosti može tumačiti kao svjestan tehnološki izbor temeljen na boljim funkcionalnim svojstvima gotovih proizvoda.

Analiza lončarije s područja Istre pokazala je da većina uzoraka sadrži fragmente glinovitih prevlaka karakterističnih za poligenetska tla tipa terra rossa. Ovi fragmenti nastaju kao posljedica pedogenetskog procesa iluvijacije, pri kojem se glinovite čestice ispiru iz gornjih (eluvijalnih) horizonata i talože u donjim (iluvijalnim) zonama, ispunjavajući pore i šupljine unutar karbonatnih stijena u kontaktu s tlom (Fedoroff, 1997; Durn, 2003; Poch, 2014; Durn *et al.*, 2023). Geokemijske značajke ovih materijala, uključujući omjere elemenata poput La/Ce, Sm/Nd i Ti/Al, pokazuju podudarnost s geokemijskim signalima terra rosse sa sjeverozapadnog dijela istarskog poluotoka (Durn *et al.*, 2023; Kudelić i Neral, 2025) te crvenih paleotala s otoka Suska (Durn *et al.*, 2018a; 2018b) što ukazuje na to da su istarski lončari koristili terra rossu kao sirovinu za izradu lončarije.

Na temelju sličnih geokemijskih zapisa između uzoraka lončarije i glinovitih sirovina prikupljenih u neposrednoj blizini istraživanih nalazišta u središnjoj Hrvatskoj i Istri, može se zaključiti da većina analizirane keramike potječe iz lokalnih izvora. Pretpostavlja se da su sirovine pribavljane u radijusu do 4–5 km od mjesta proizvodnje (Neral *et al.*, 2024; Kudelić i Neral, 2025), što je u skladu s opažanjima prethodnih etnoarheoloških istraživanja (Arnold, 1980:149; 1985:39–49; Gosselain i Livingstone Smith, 2005) koja upućuju na to da su lončari težili minimalizirati vremensko i energetska ulaganja prilikom nabave primarnih sirovina.

Unatoč prevladavajućoj uporabi lokalno dostupnih sirovina, rezultati istraživanja ukazuju na prisutnost i nekoliko uzoraka lončarije izrađenih od sirovina nelokalnog podrijetla. Iako rijetki, takvi nalazi pružaju važne indikacije o mogućem utjecaju dodatnih čimbenika, uključujući trgovinske veze, migracijske procese ili druge oblike socioekonomskih interakcija. Primjerice, lončarija iz neolitika iz središnjeg dijela Hrvatske pokazuju različite koncentracije elemenata rijetkih zemalja u usporedbi s ostatkom analiziranog materijala (Neral *et al.*, 2024). Ova odstupanja mogu upućivati na nabavu sirovina s udaljenijih lokacija ili na distribuciju sirovina ili gotovih proizvoda unutar neolitičkih zajednica. U lončariji s istarskih nalazišta zabilježena prisutnost rekristaliziranih planktonskih foraminiferskih kućica (Kudelić i Neral, 2025). Ovi mikrofosili tipično se nalaze u laporima, a njihova prisutnost u kontekstu geološke građe ukazuje na mogući transport lapora kao sirovine iz unutrašnjosti Istre do arheoloških nalazišta (Ćosović *et al.*, 2004; Babić *et al.*, 2007; Gulam *et al.*, 2014).

Uz glinoviti materijal kao osnovnu sirovinu, rezultati provedenog istraživanja upućuju na to da gotovo svi analizirani uzorci keramike sadrže dodane primjese. Njihova prisutnost čini se funkcionalno uvjetovanom, pri čemu je izbor primjesa često bio povezan s namjenom keramičke posude. Naime, posude namijenjene kuhanju, koje su bile izložene ponavljanim termičkim opterećenjima, redovito sadrže primjese. Nasuprot tome, manje keramičke posude poput šalica, za koje se pretpostavlja da su se primarno koristile za posluživanje ili pohranu, češće su izrađivane bez dodatka primjesa (Neral *et al.*, 2023). Ovakvi rezultati upućuju na to da je uporaba primjesa prvenstveno bila motivirana potrebom za poboljšanjem fizičko-mehaničkih svojstava sirovinke smjese i gotovog proizvoda, uključujući bolju obradivost, veću toplinsku provodljivost te otpornost na termički šok. Ipak, u određenim kontekstima i slučajevima, na odabir vrste primjesa utjecali su i čimbenici povezani s tradicijskim praksama (Gosselain, 1999; Kreiter, 2007; Larsson, 2009: 351–355; Pikirayi & Lindahl, 2013; Kudelić *et al.*, 2018; Holmqvist *et al.*, 2018; Quinn, 2022: 223).

Analiza lončarije ukazuje na znatne razlike u vrstama korištenih primjesa kroz različita razdoblja prošlosti, kao i među pojedinim geografskim regijama. Utvrđeno je korištenje različitih vrsta primjesa, uključujući pijesak, litoklaste, kalcit, grafit, biljne ostatke poput pljeve, fragmente mekušaca te grog (usitnjena keramika). U prapovijesnim razdobljima – od neolitika do željeznog doba – prisutnost određene vrste primjese povezana je s pojedinim razdobljima prošlosti, što je u skladu s karakteristikama kućne ili lokalno organizirane proizvodnje lončarije. Takva je

proizvodnja bila čvrsto ukorijenjena u društveni okvir i usmjerena na zadovoljenje potreba zajednice. Nasuprot tome, u povijesnim razdobljima (rimsko doba, kasna antika i srednji vijek) zabilježen je značajan pomak prema standardizaciji proizvodnje, koja je prelazila okvire lokalnih zajednica. Time je lončarska proizvodnja poprimila obilježja neki oblik industrijalizacije, usmjerene na učinkovitost i skraćivanje proizvodnog procesa, pri čemu lončarija i korištene sirovine sve manje odražavaju karakteristike kućanstava ili zajednica. Osim vremenskih razlika, uočene su i regionalne varijacije u korištenju primjesa. U kontinentalnoj Hrvatskoj evidentirana je veća raznolikost dodanih materijala u odnosu na područje Istre, gdje se kroz gotovo sva istražena razdoblja kao dominantna primjesa javlja kalcit. Takav obrazac snažno upućuje na presudnu ulogu okoliša u oblikovanju tehnoloških odluka prilikom proizvodnje lončarije. U istarskom krškom krajoliku, kalcit se prirodno nalazi unutar karbonatnih stijena, a njegova niska tvrdoća i izvrsna kalavost omogućuju jednostavno usitnjavanje i dodavanje glinovitoj smjesi. Nadalje, kalcit posjeduje koeficijent toplinske ekspanzije sličan onome glinovitog materijala (Rye, 1976; Broekmans *et al.*, 2004; Shoval *et al.*, 2006), čime se smanjuje rizik od pucanja tijekom sušenja i pečenja, što dodatno poboljšava tehnička svojstva keramičkih posuda. Ove okolnosti upućuju na to da su lončari prepoznavali i svjesno koristili tehnološke prednosti kalcita kao lokalno dostupne i lako obradive sirovine.

Analizom temperatura i atmosfera pečenja keramike jasno su se utvrdile razlike u karakteristikama proizvodnje između prapovijesnih i povijesnih razdoblja. Dobiveni rezultati upućuju na to da je većina prapovijesne keramike bila pečena na temperaturama nižim od 800 °C, u uvjetima nepotpune oksidacije. Ovi podaci ukazuju na ograničenu kontrolu nad termičkim parametrima tijekom procesa pečenja te ukazuju da se pečenje najčešće odvijalo na otvorenoj vatri ili u plitkim jamama (Kudelić i Sirovica, 2022: 157–165; Neral *et al.*, 2023). Unatoč ovim obrascima, identificirane su i određene iznimke. Povremena prisutnost visoko-temperaturnih mineralnih faza poput kristobalita (Neral *et al.*, 2023; Kudelić i Neral, 2025) i spinela (Neral *et al.*, 2024), sugerira da su u pojedinim slučajevima temperature pečenja dosežale ili nadilazile 1000–1100 °C (Cultrone *et al.*, 2001; Cultrone i Carrillo Rosúa, 2020). Međutim, s obzirom na njihovu ograničenu učestalost u analiziranim uzorcima, ove pojave vjerojatno ne odražavaju ustaljene tehnike pečenja, već se mogu protumačiti kao rezultat sekundarnog izlaganja visokim temperaturama, primjerice uslijed požara ili drugih izvora nenamjernog toplinskog djelovanja.

Od mlađeg željeznog doba nadalje zabilježena je značajan tehnološka promjena u metodama pečenja, koji uključuje široku primjenu peći. Ova promjena omogućila je postizanje znatno viših temperatura, kao i djelomično ili potpuno redukcijske atmosfere tijekom pečenja. U tom kontekstu, sve češća pojava visoko-temperaturnih mineralnih faza te povremena vitrifikacija matriksa u uzorcima kasnoantičke i srednjovjekovne keramike iz središnje Hrvatske (Kudelić *et al.*, predano; Neral *et al.*, 2024) upućuju na termalne uvjete u rasponu od 800 do 1100 °C. Ova tehnološka inovacija odražava kontrolu nad procesom pečenja što dodatno ukazuje na prijelaz prema standardiziranoj i specijaliziranoj proizvodnji keramičkih proizvoda, u skladu s društveno-ekonomskim transformacijama karakterističnima za ta povijesna razdoblja.

KEYWORDS

Clay

Tempering materials

Physical and mechanical properties

Provenience

Ancient pottery technology

Archaeometry

KLJUČNE RIJEČI

Glina

Primjese

Fizička i mehanička svojstva

Podrijetlo

Drevna lončarska tehnologija

Arheometrija

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1. Introduction

Ceramics represent the earliest synthetic material created by humans (Rice, 1987). The production process involves the firing of clay (from the Greek *keramos*, meaning "fired substance"), which induces physical and chemical transformations, resulting in a newly formed, highly durable material. Since its invention, ceramics have been an indispensable component of ancient households. Furthermore, it is often the only remnant of the material culture that constitutes the evidence for interpreting ancient human history.

The most common and fundamental approach in the archaeological analysis of pottery involves examining the morphological and stylistic characteristics of vessels, which primarily serve as chronological indicators and aid in establishing the spatial and temporal context of artefacts and archaeological sites. However, the study of pottery from a materials science perspective—encompassing the analysis of raw materials such as clays and tempering materials—provides a broader spectrum of scientifically grounded data. This approach facilitates the reconstruction of technological processes thereby contributing to a deeper understanding of the technologies employed by past communities. Such research is multidisciplinary, necessitating collaboration across multiple scientific disciplines—including natural, technical, and human sciences—and fields such as archaeology, geology, and geological engineering to achieve a comprehensive understanding of ancient societies. Following this multidisciplinary approach, this doctoral research examines the raw materials and pottery production technologies utilized by communities across various historical periods in Croatia. The study primarily employs geological and geological engineering methods applied to both archaeological and geological materials to analyse pottery raw materials and production techniques.

This research comprises pottery from three case studies, each situated in distinct geographical and geological regions (Fig. 1) shaped by diverse socio-economic and cultural influences over the history. The first case study examines the Baranja region, focusing on the archaeological site of Jagodnjak-Krčevine in Eastern Croatia. The second case study centres on the Turopolje region, specifically the Staro Čiče-Gradišće site in Central Croatia, supplemented by data from the nearby Kurilovec-Belinščica site. The third case study investigates the northern part of the eastern Adriatic coast, with emphasis on the Istrian region. To ensure a representative

archaeological sample encompassing different historical periods, three sites within close proximity (no more than 3 km apart) were selected: Old Rakalj, Šiljar, and Saint Theodore.

The archaeological sites and associated pottery analysed in this study are multi-period, spanning from the Neolithic to the Late Medieval period, allowing for an examination of temporal variations in raw material selection. However, these prehistorical and historic periods were not contemporaneously represented across all regions of present-day Croatia. Furthermore, the investigated archaeological sites exhibit discontinuous patterns of occupation, indicative of shifting settlement dynamics and the selective utilization of specific locations throughout time. Nevertheless, the investigated multi-period sites contain ceramic material corresponding to multiple distinct phases of habitation, as illustrated in Fig. 2. In cases where no material evidence from a specific phase of occupation was documented at a given site, supplementary data from methodologically comparable studies conducted in the respective region were integrated to enhance the analytical framework. Additionally, to provide a comparative analysis between archaeological ceramics and locally available raw materials, and to investigate the provenience of these materials, the study includes an examination of clayey deposits and intentionally added tempers. These materials were systematically collected from areas in proximity to the archaeological sites under investigation. In the context of this doctoral thesis, the term provenience is employed to denote the geographic origin of the raw materials used in ceramic production, in accordance with the distinction articulated by Price & Burton (2011). While provenance refers to “an artifact’s résumé,” encompassing its post-depositional history and contextual associations, provenience specifically designates “an artifact’s birthplace,” or its original geological source.

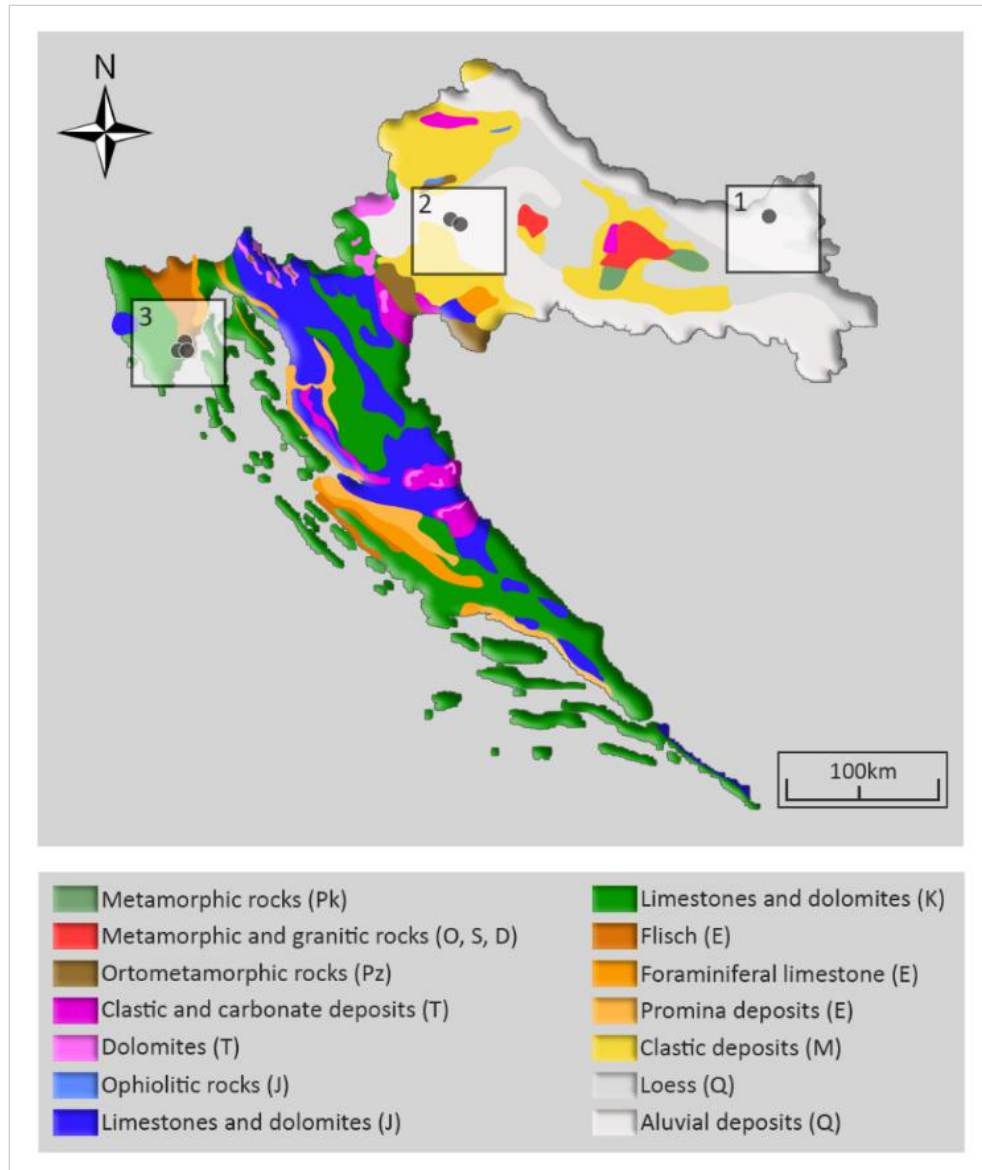


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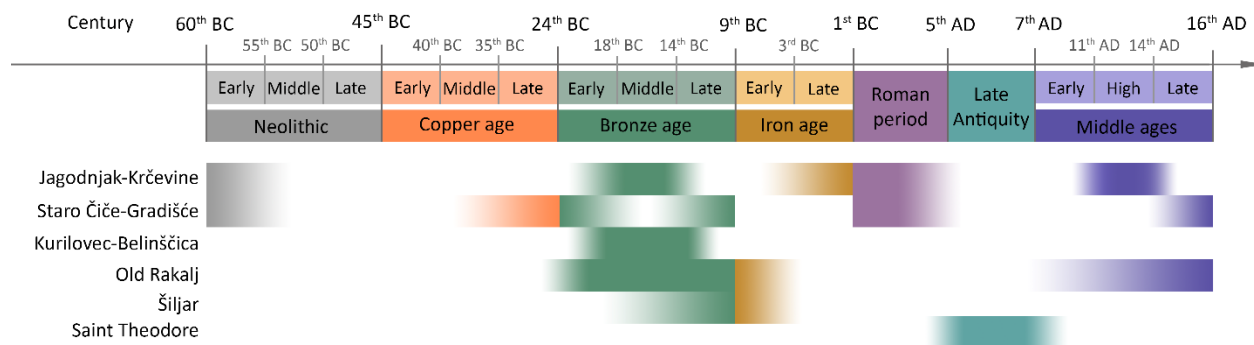


Figure 2. Chronological representation of the occupation of investigated archaeological sites

1.1. Previous studies

The analysis of clays and clay-rich materials in Croatia is predominantly conducted within the fields of natural and technical sciences. For example, research on the characteristics (Gverić *et al.*, 2020) and hydro-mechanical properties of bentonite clays has been carried out to assess their suitability for use in radioactive waste disposal sites (Domitrović *et al.*, 2012). Additionally, pelitic sediments, including clay fractions, have been analysed to reconstruct paleoenvironmental conditions (Avanić *et al.*, 2021) and to investigate the provenance of materials deposited within the Pannonian Basin system (Grizelj *et al.*, 2017). Clay minerals have also been extensively examined in paleosols (terra rossa) to provide insights into the paleoenvironmental and paleoclimatic conditions of their formation (Durn *et al.*, 2014; Durn *et al.*, 2018a; Durn *et al.*, 2018b; 2021; 2023; Perković *et al.*, 2024). Furthermore, research on the reactivity of clay minerals has been conducted to explore their potential application in cement and brick production (Kovačević, 2015; Flegar *et al.*, 2019, 2020, 2023; Ram *et al.*, 2023). In these and numerous other studies, clay is primarily considered as a sedimentary deposit formed within specific paleoenvironmental conditions, with a focus on its mineralogical and geochemical composition, diagenetic alterations, and potential for industrial use.

Conversely, studies that investigate clay as a raw material for ancient pottery production in Croatia remain relatively scarce (Kudelić *et al.*, 2018; Miše *et al.*, 2019, 2020; Hawkins *et al.*, 2025). More common are studies focused on ancient pottery technology, though these are primarily conducted on archaeological materials (e.g. Šegvić *et al.*, 2011; Miloglav, 2013, 2016; Miloglav *et al.*, 2015; Kudelić, 2017; Spataro, 2019; Kudelić & Mileusnić, 2020; Miše & Quinn, 2022). However, these investigations are often fragmented, without a standardized methodological

framework for material processing and analysis, and typically focus on individual archaeological sites or specific historical periods. Significant progress in archaeological research has been made through the Croatian Science Foundation project Technological Features and Cultural Practices in Prehistoric Pottery Traditions in Croatia (UIP-2020-02-3637, acronym: prePOT). This project adopts a comprehensive approach to the study of pottery traditions, encompassing diverse geographical regions and cultural groups over an extended chronological framework, thereby offering a broader and more systematic understanding of pottery production and use in the past.

1.2. Raw materials and its properties

Pottery raw materials encompass clay-rich materials, which serve as the fundamental component of all ceramic products, and tempers—additives deliberately incorporated into the clay. The clay-rich materials used in ceramic vessel production typically include sandy clays, silty clays, and clays, each exhibiting specific properties essential for suitability in ceramic manufacturing. However, the most important characteristic of clayey materials is the presence of clay minerals mainly originated by the weathering of feldspar or by low temperature hydrothermal alteration of crystalline rocks (Montana *et al.*, 2011). Clay minerals are hydrous silicate minerals belonging to the phyllosilicate group and are typically characterized by their fine particle size, generally smaller than 2 μm , and their distinctive sheet-like structure. These minerals primarily consist of silica, alumina, magnesium, oxygen, and water. In the context of pottery raw materials, a critical property of clay minerals is their capacity to retain various forms of water, classified based on the nature of its bonding to the clay particles. This property is integral to the functionality of clays, enabling them to be shaped, dried, and subsequently fired into durable materials—a key factor in their use as a primary resource for ceramic vessel production over millennia. Clay minerals are broadly classified into two categories: swelling (e.g. smectite, vermiculite) and non-swelling (e.g. kaolinite, chlorite, illite) types. While all clay minerals exhibit adsorbed water on their surfaces, swelling clay minerals possess a distinctive ability to absorb water into their interlayer spaces, resulting in volume expansion upon hydration (Velde, 1992; Velde & Meunier, 2008; Montana *et al.*, 2011; Druc & Velde, 2017). However, a high overall content of clay minerals, particularly swelling clay minerals, significantly increases the total volume of the clay upon hydration, posing considerable challenges for potters during vessel production.

Therefore, to address the challenges associated with excessively high plasticity and to enhance the mechanical properties of the vessels, potters intentionally incorporated various non-plastic materials, known as tempers, into the clay matrix. These materials enhance the workability of the clay, reduce shrinkage and plasticity, and improve the vessel's thermal shock resistance and heating efficiency (Skibo *et al.*, 1989; Hoard *et al.*, 1995; Vekinis & Kilikoglou, 1998; Velde & Druc, 1999; Kilikoglou *et al.*, 1995, 1998; Hein *et al.*, 2008; Müller *et al.*, 2010, 2015; Albero Santacreu, 2014). The most common types of utilized tempering materials can be classified into three broad categories: mineral tempers (e.g., calcite, quartz, graphite, and fragments of sedimentary, metamorphic, and igneous rocks), organic tempers (e.g., shells, bones, and plant-based materials), and anthropogenic temper (crushed ceramics or grog). Beyond their contribution to the physical and mechanical properties of the ceramics, the selection of tempering materials is often characteristic of particular cultures or historical periods. As such, temper analysis provides valuable insights for dating ceramic artefacts and understanding trade relationships (e.g. Rice, 1987; Kreiter, 2007; Holmqvist *et al.*, 2018).

1.3. Pottery technology

Pottery production is a multifaceted technological process comprising several stages, collectively conceptualized within the framework of the *chaîne opératoire* (Leroi-Gourhan, 1964). This framework encompasses the entire manufacturing sequence, defined as a series of operations that transform raw materials into finished products. Each of these operations, or stages, is characterized by specific techniques that are inherently linked to the societies that developed them (Roux, 2019). As such, they reflect the dynamic interplay between humans, craftsmanship, and material culture. The technological process begins with the selection of raw materials, followed by the preparation of clay paste, the shaping of the vessels, drying, firing, and, ultimately, the use of the finished products. Each stage involves significant complexity and variability, necessitating detailed investigation to uncover the techniques employed in ancient ceramic production (Albero Santacreu, 2014).

1.3.1. Selection of raw materials

The technological process of pottery production begins with the selection of raw materials, including both clayey and tempering materials, a critical step that directly affects the quality and performance of the final ceramic products. The choice and use of materials are primarily influenced by local geological characteristics, availability, technical constraints, and functional needs (e.g. Rye, 1981; Gosselain & Livingstone Smith, 2005; Michelaki *et al.*, 2014). This doctoral research aims to investigate these factors to better understand the selection and utilization of pottery raw materials in the past. However, these choices are also in relation with traditional practices, and the symbolic meanings ascribed to certain materials (Arnold, 2000; Livingstone Smith, 2000; Gosselain & Livingstone Smith, 2005).

Archaeological and ethnographic research suggests that communities predominantly sourced clay-rich materials from areas near their settlements or production sites. As ceramic production was an integral part of daily life, potters primarily crafted vessels to meet the needs of their own communities. Consequently, given that clay was the primary raw material, potters prioritized the use of readily available local resources, thereby minimizing the time and effort required for material collection (e.g., Arnold, 1978; Rice 1987: 116, Gosselain & Livingstone Smit., 2005; Albero Santacreu & Mateu Vicens, 2012; Roux, 2019: 23).

The methods of clay extraction varied depending on the local environment and geology, including surface collection, pit excavation, gallery extraction, and underwater harvesting. Surface collection involved gathering raw materials from just beneath the surface, often on flat ground, slopes, or hillsides, without the need for excavation. Pit extraction required digging vertically or at an angle to reach the appropriate clay layer, with pits typically ranging from 1 to 2 meters in depth and 2 to 3 meters in diameter. Gallery extraction entailed digging a vertical shaft to access the clay bed, then extending it horizontally to gather more material. Finally, underwater extraction involved retrieving clay from submerged deposits, such as riverbeds (Gosselain & Livingstone Smith, 2005).

The selection of tempering materials, like that of clayey raw materials, was primarily influenced by the geological characteristics of a given area and aimed at optimizing the functional properties of ceramic products. However, research has also shown that the choice of specific tempers was culturally driven (Arnold, 2000; Gosselain & Livingstone Smith, 2005; Kreiter *et al.*, 2007; Michelaki *et al.*, 2014; Albero Santacreu *et al.*, 2014). Consequently, distinguishing between

naturally occurring clayey raw materials and intentionally added tempers is crucial for understanding both the technological strategies employed in pottery production and other considerations that influenced raw material selection.

The choice of raw materials plays a crucial role in determining the quality, durability, and overall performance of ceramic vessels. Therefore, to fully understand the rationale behind the selection of specific clays and tempers, it is crucial to conduct a detailed examination of their physical properties, provenience, and their impact on the strength and functionality of the final product. This can be accomplished through the application of petrographic, mineralogical, and geochemical analyses, with particular emphasis on rare earth element concentrations. Together, these methods offer comprehensive insights into the sources of raw materials and their defining characteristics, contributing to a deeper understanding of technological choices in ceramic production.

1.3.2. Paste preparation

Following the selection and procurement of raw materials, potters undertake the preparation of the clay paste used in ceramic vessel production. This process typically involves the removal of larger impurities to refine the clay's composition, as well as the addition of specific tempering materials to improve its mechanical and thermal properties (Rye, 1981). In some cases, the preparation also includes drying the clay and crushing it into smaller lumps prior to paste formation.

The specific methods employed for preparing the clay depend largely on the type and properties of the raw material. Certain clayey materials, particularly those with adequate shaping ability and those obtained in a suitably moist state, can be utilized directly from their natural source with little to no modification. In such cases, only minimal refinement is required, such as the removal of coarse inclusions or vegetal fragments. Conversely, clays characterized by an extremely fine texture and high plasticity often require modification through the intentional addition of tempering materials. This adjustment modifies the properties of the raw clay by enhancing the clay's suitability for pottery production and addressing issues such as excessive plasticity and improving its workability (Rice, 1987).

Potters were required to properly process and prepare tempering materials before incorporating them into the clay. The selection of tempering materials was often associated with the intended function of the vessels (Arnold, 2000). Specifically, research has demonstrated that

potters primarily added tempering materials to ceramic vessels intended for cooking, as these materials enhanced thermal shock resistance and improved heating efficiency (Rye, 1981: 27; Schiffer & Skibo, 1987; Müller *et al.*, 2010; 2014). In contrast, fine tableware, such as cups and smaller vessels that were not subjected to high temperatures, was more frequently produced without the addition of tempering materials (Arnold, 2000). The inclusion of tempering materials helps to ensure that the ceramic maintains structural integrity during thermal cycles by matching the thermal expansion rates of the clay matrix, thus preventing fractures due to dilatometric discrepancies during repeated heating and cooling (Albero Santacreu, 2014: 154). As a result, tempered ceramics are less prone to strength reductions after thermal shock compared to non-tempered ceramics (Schiffer & Skibo, 1987).

Throughout history, potters in the territory of Croatia and neighbouring regions have employed a diverse range of tempering materials to enhance the properties of clay and the resulting ceramic products. Commonly used mineral tempers include calcite, graphite, sand, and fragments of sedimentary, metamorphic, and igneous rocks (e.g. Waldhauser, 1992; Dizdar, 2001: 87; Žibrat-Gašparič, 2004; Mange & Bezecky, 2006; Dolenec (Kramar) *et al.*, 2012; Spataro & Meadows, 2013; Spataro, 2019; Kreiter *et al.*, 2014; Drnić & Miletić-Čakširan, 2014; Kudelić, 2017; Cannavó & Levi, 2018; Marton *et al.*, 2020; Germinario *et al.*, 2020; Grifa *et al.* 2021; Maritan *et al.*, 2009; 2021). Additionally, certain communities employed grog (ceramoclasts) as a tempering material (e.g. Spataro, 2002; Kreiter, 2007, Maritan *et al.*, 2009; Miloglav, 2016; Kudelić, 2016, 2017; Žibrat-Gašparič & Dolenec, 2015; Spataro, 2019), consisting of crushed ceramic fragments derived from broken vessels, hearths, daub, weights, or other ceramic objects. Organic tempers, such as shells and plant-based materials, were also incorporated into pottery production (e.g. Spataro & Meadows, 2013; Spataro, 2019; Tenconi *et al.*, 2013; Kreiter *et al.*, 2013; 2014; Žibrat-Gašparič & Dolenec, 2015; Marton *et al.*, 2020).

The method of temper preparation can vary depending on the specific type of material used. However, most tempering materials require crushing and sieving, with the desired particle size of the inclusions determined by the wall thickness and the forming technique employed. Once collected and properly prepared, the tempers were mixed with either wet or dry clay to create a workable paste (Rye, 1981: 27).

A comprehensive examination of the relationship between the primary raw material (clay) and tempering materials is essential for understanding the impact of tempers on the physical and

mechanical properties of ceramics. Additionally, such an analysis provides insight into the underlying factors influencing the selection and utilization of these materials, which result from a complex interplay of local resource availability, the intended function of ceramic vessels, and potentially culturally determined practices.

1.3.3. Shaping

After preparing the clay paste, potters proceeded with shaping the material into ceramic vessels. The process of vessel formation can be broadly categorized into two primary techniques: hand-building and wheel-throwing (Rye, 1981: 62; Rice, 1987: 124–132). Each of these categories encompasses multiple sub-techniques, which vary depending on the size, shape, and intended function of the vessel. Hand-building refers to the manual construction of a vessel without the use of additional tools. It represents the simplest and most ancient ceramic-forming technique, extensively utilized throughout prehistory (Rice, 1987: 124–132; Quinn, 2022: 241). In contrast, wheel-throwing involves shaping the vessel on a rotating potter's wheel, requiring a different approach to clay preparation. The clay paste used for wheel-thrown vessels required a higher moisture content and greater plasticity, as the drawing action necessary to raise the vessel walls would be difficult to achieve with stiff clay. Furthermore, the increased air circulation during wheel rotation facilitated the rapid evaporation of water, accelerating the drying process. Additionally, the paste used for wheel-throwing was typically finer in texture and without tempering materials to reduce abrasion on the potter's hands during shaping (Rice, 1987: 128–129; Quinn, 2022: 241–244). Potters had to carefully adjust the properties of the clay paste to align with the specific forming technique, as the use of an unsuitable paste could hinder the successful formation of a complete vessel.

Consequently, shaping techniques and their associated constraints must be considered, as they directly influence raw material selection and preparation, reflect technological advancements and potters' expertise, and determine the overall efficiency of the manufacturing process.

1.3.4. Drying

Drying is an important phase in the ceramic production process that follows the shaping of vessels. During this stage, a significant reduction in both volume and weight occurs as water evaporates from the surface of the clay minerals. Proper drying is essential, as excessively rapid moisture loss or incomplete drying can lead to structural defects, which may compromise the vessel's integrity

either during drying itself or later during firing. The duration of the drying process varies from several days to several weeks, depending on factors such as wall thickness and the inherent properties of the clay. Thinner vessel walls facilitate faster drying, whereas thicker walls prolong the process. Additionally, clays with a higher concentration of clay minerals retain more water, necessitating extended drying periods. In contrast, pastes with a greater proportion of non-plastic tempers dry more quickly and can be exposed to direct sunlight without a significant risk of flaw formation (Rice, 1987: 152–153; Velde & Druc, 1999: 154; Albero Santacreu, 2014: 80). Given these variations, careful regulation of the drying process is necessary to maintain the structural integrity and overall quality of the final ceramic product.

Studying the properties of different paste recipes is essential for understanding the duration and complexity of the drying process within the broader technological framework of ceramic production. Examining the relationship between clay composition, tempering materials, and drying behaviour provides insight into the extent to which ceramic durability and deformation are influenced by different raw material combinations.

1.3.5. Firing

One of the most significant properties of clay is its ability to undergo irreversible hardening when exposed to heat. This transformation, which results in the production of durable ceramics, is governed by three primary factors: time, temperature, and atmosphere (Gosselain, 1992; Velde, 1992; Druc & Velde, 2017: 111, 124). Firing methods, while varying significantly across societies, can generally be categorized into two fundamental techniques: firing without a kiln and firing with a kiln. The earliest method involved firing ceramics in an open fire or bonfire, characterized by its relatively short duration (typically less than one hour), lower firing temperatures (usually below 800 °C), and an uncontrolled atmosphere (Rye, 1981: 98; Rice, 1987: 153–158). Over time, potters began to employ various types of kilns, which, despite the labour-intensive and time-consuming process of construction, offered significant advantages. Kilns offered enhanced control over the firing process and atmosphere, enabling the attainment of higher temperatures, and more efficient utilization of fuel resources (Rice, 1987: 152–162). The identification of the firing method can be determined by analysing the colour variations in ceramic cross-sections (Rye, 1981: 114–117).

Regardless of the firing technique employed, all firing processes induce significant mineralogical and structural transformations within the clay, which can be analysed by examining

ceramics under a polarizing microscope (ceramic petrography) and determining their mineralogical composition using X-ray powder diffraction (XRPD) methods. These changes commence at relatively low temperatures (up to 300 °C) as an extension of the drying process. During this stage, water molecules adsorbed onto the surfaces of clay particles volatilize, leading to weight loss and material shrinkage. As the temperature rises, the chemically bonded water within the interlayer spaces of clay minerals begins to be expelled, a process that occurs over a broad temperature range, approximately between 300 and 800 °C, and is largely determined by the specific types of clay minerals present. During this stage, the release of intralayer water induces significant structural alterations within the clay minerals. Concurrently, organic materials present in the clay undergo oxidation, releasing carbon dioxide (CO₂). As firing progresses to higher temperatures (800–1000 °C), the minerals within the ceramic body, whether originating from the clay matrix or added as tempers, begin to decompose, leading to the formation of new mineral phases such as lime, hematite, cristobalite, and mullite (Gruver, 1950; Cultrone *et al.*, 2001; Nodari *et al.*, 2007; Cultrone & Carrillo Rosua, 2020). At very high temperatures, exceeding 1000 °C, sintering and vitrification processes occur. These processes produce a glassy, non-porous ceramic body, marking the final stage of transformation within the material (Maniatis & Tite, 1981; Cultrone *et al.* 2001).

Assessing the firing temperatures and methods utilized by different communities is essential for achieving a more comprehensive understanding of the ceramic production process and for identifying distinctive patterns of firing techniques associated with specific historical periods or geographic regions.

1.4. Methodology

The research methodology implies the application of various analytical methods that enable the reconstruction of the technological process of pottery production and an insight into the characteristics and provenience of raw materials used in the past. The research began with field sampling of potential clayey raw materials near investigated archaeological sites within a radius of up to 10 km from the site based on earlier research and the assumed distance that potters travelled to obtain raw materials (Arnold, 1980: 149). Subsequently, the collected clayey samples were shaped into briquettes and fired in a laboratory kiln for 60 min at 650 °C with 30 min of firing

at the maximum temperature (soaking time). These firing conditions were selected to replicate the presumed firing conditions of prehistoric pottery production (Maniatis & Tite, 1981).

Archaeological pottery and experimental ceramic briquettes were analysed by optical microscopy of thin sections using a polarizing microscope. The method enables the collection of data on the mineralogical and petrographic composition of the samples, matrix microtexture, paste recipes and the estimation of the firing temperature. The pottery sherds were categorized into petrographic fabric groups for each archaeological site based on variations in the mineral composition and texture of the clay matrix, which provided insights into the different types of raw clay materials used. Furthermore, the fabric groups were further subdivided into fabric subgroups according to specific characteristics of the mineral composition, size, quantity, roundness, and distribution of grains within the matrix. These distinctions were indicative of the presence of materials intentionally added as tempers (PCRG, 2010; Quinn, 2013). Comparison of the mineralogical composition and matrix texture of archaeological pottery samples and experimental ceramic briquettes enabled determination of the clay raw materials used for the production of ceramic vessels.

Furthermore, the mineralogical composition of ceramic and clayey samples was analysed using X-ray powder diffraction (XRPD). In addition to offering detailed insights into the mineralogical composition of ceramic and clay samples, XRPD analysis enabled the estimation of firing temperatures by identifying mineral phases that form within specific thermal ranges. Furthermore, the $<2\ \mu\text{m}$ fraction was isolated from the clayey samples and subjected to a series of treatments, including K and Mg saturation, ethylene glycol solvation, dimethyl sulfoxide solvation, and heating to 350 °C and 650 °C. These procedures were conducted to facilitate the identification of individual clay minerals, following the methodology outlined by Moore & Reynolds (1997).

Atterberg plasticity limits (liquid and plastic limit, and plasticity index) were determined for selected clayey samples, and sediment classification was refined using laser granulometry. To achieve a more comprehensive insight into the provenience of clay materials used in the past, geochemical analyses were conducted, including inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma emission spectrometry (ICP-ES), on both archaeological pottery and clayey samples. The resulting geochemical data were statistically processed using bivariate and multivariate analytical methods. Furthermore, to assess variability in technological processes and the mechanical properties of ceramics produced from different clay

types and tempering materials, an experimental study was conducted. The strength index of experimental ceramic samples was evaluated using the point load test (PLT). The obtained results were subjected to statistical analysis to evaluate variations within the analysed sample sets.

1.5. Objectives and hypotheses of the research

Main objectives of this research are: (1) To determine the type, provenience and individual physical and mechanical properties and availability of the potential raw materials used for pottery production during different periods of the past in Croatia, (2) to conduct a comparative analysis of archaeological ceramics and pottery raw materials collected in the environment, (3) to determine the characteristics of the technological procedures (clay paste preparation and firing techniques) for each investigated period of the past and (4) to investigate the variability in the choice of the raw materials and pottery technology through different periods of the past.

These objectives were based on following hypotheses: (1) Clayey raw material used for pottery production was supplied in the immediate vicinity of the settlement where the pottery was produced, (2) the choice of pottery raw materials is conditioned by its availability and the geological structure of the area and (3) technological procedures of pottery production (clay paste preparation and firing techniques) are not necessarily conditioned by the optimization of the technological solutions.

1.6. Scientific contribution

Examining the characteristics of the pottery raw materials and the technological procedures will contribute to understanding the variabilities in the choice of the raw materials and pottery technology through different periods of the past in Croatia. The research will thus consider the ancient technologies and the dynamics of landscape use through a broad spatio-temporal context i.e., in different areas and within cultural groups from different periods of the past in Croatia.

2. Original scientific papers

Paper 1

Neral, N., Kudelić, A., Maričić, A., Mileusnić, M. (2023) Pottery technology through time: Archaeometry of pottery and clayey raw material from the multi-period site in eastern Croatia. The Mining-Geology-Petroleum Engineering Bulletin, 38(1), 1–21.doi.org/10.17794/rgn.2023.2.1

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Original scientific paper



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Abstract

Consideration of multiperiod archaeological sites to understand the mechanisms of large-scale cultural changes is still a very rare research topic in Croatia. Technological traditions are of great importance, especially in the context of considering continuity, innovation, and change. In this paper, we used an archaeometric approach to investigate pottery technology. Therefore, this article presents an analysis of petrography and mineralogy of archaeological ceramics and potential raw materials collected in the vicinity of the multi-period archaeological site (the Neolithic through the Medieval period) Jagodnjak-Krčevine located in eastern Croatia, i.e. the south-western part of the Pannonian Basin. The primary goal is to determine what kind of clay recipe (clay and temper) potters used to make vessels in order to better understand their variability in the context of techno-functional features. The additional objective is to examine the availability and quality of clays for pottery production and to study their distribution in the local landscape. The analytical methods applied in the research are optical microscopy, X-ray diffraction, and grain size analysis. The comparison of archaeological ceramics with clayey materials established that ancient potters used locally available clays, and the selection of temper material added to the clay represents a strong element of techno-tradition, which is more pronounced for prehistoric communities. Furthermore, these results represent the first research in Croatia focused on a discussion about distances that potters travelled to obtain their resources in the different periods of the past, which can contribute to the study of a regional system of production and landscape use.

Keywords:

archaeometry; Baranja; clayey raw material; field sampling; pottery technology

1. Introduction

Raw materials are an essential component of every production process and natural materials represented the only raw materials available in ancient times. In the context of the production process, pottery-making was an integral part of the evolution of human culture since the Neolithic and is considered one of the most important components of tradition and technological development. While manufacturing techniques have not changed significantly, especially for prehistory, diversity in morphology and style in archaeology represent a very significant and usually the main indicator of chronology and cultural determination. However, subtle changes and shifts in technology (or just techniques) affect the appearance and performance of the final product, and the reasons for such changes can be triggered by various

factors, such as social or, for example, environmental changes. Therefore, investigating past technologies throughout the reconstruction of that process necessarily implies looking at each step in the operational sequence (van der Leeuw, 1984; Sillar and Tite, 2000; Albero Santacreu, 2014: 53–59) from procurement of the raw material, paste preparation, shaping, drying, and firing (Hunt, 2016; Roux, 2019) in order to not only reconstruct that process, but also to try to understand what is in the background of such changes. To investigate these processes, archaeometry has been increasingly considered as the best starting point for study (Tite, 1999), especially the choices of potter raw material used diachronically over a long period of time.

To obtain a reliable dataset of potential clayey sources, it is necessary to conduct field sampling near the location of the ancient settlement. Although in most studies it is assumed that the raw material is of local origin (Arnold, 1978; Gosselain et al., 2005; Albero Santacreu and Mateu Vicens, 2012; Michelaki et al., 2012;

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Figure 1: A selection of pottery chosen for analysis from the archaeological site Jagodnjak-Krčevine: a, b (Neolithic, samples 8987, 8988); c, d (Bronze Age samples 9459, 9460); e, f (Late Iron Age, samples 8991, 9000); g, h (Roman period, samples 8997, 9001); i, j (Medieval, samples 8996, 8752)

De Bonis et al., 2013; Žibrat Gašparič and Dolenec, 2015; Kudelić et al., 2018), the field sampling will give answers to the questions regarding which part of the landscape the potters either preferred or did not prefer for raw material exploitation, but also how the people oriented themselves in that landscape (Michelaki, 2014). Furthermore, it is assumed that the raw material preparation and the choice of tempering material (clay paste recipes) also reflect temporal and cultural differences in pottery practices.

This paper presents the first study on the continuity and variability of pottery technologies over more than 7000 years in Croatia according to the archaeological records from a multi-period (Neolithic, Bronze Age, Late Iron Age, Roman, and Mediaeval period) site of Jagodnjak-Krčevine located in the Baranja region. The objective is to examine the characteristics of raw materials used in the past, to determine ceramic technological features, and to study the availability and distribution of pottery raw materials in the local landscape. The aim of the study is to answer the questions of whether the ceramics from the Jagodnjak site were produced using locally available clays and whether potters from different periods had preferences for particular clay sources or clay characteristics. Moreover, the aim is to uncover the clay recipes and temper material used to make the vessels in order to understand the variability and preferences of ancient potters. To methodically address all research questions, the study focuses on the petrographic and mineralogical analyses using several analytical techniques: optical microscopy, X-ray diffraction (XRD), and additionally grain size analysis and physical properties of the collected sediments.

2. Archaeological setting

Baranja is a historical and geographical region in eastern Slavonia bordered by the Drava River in the

southwest, the Danube River in the east, and the Croatian-Hungarian border in the north. Geomorphologically, it is a lowland with a slightly raised ridge that has always been suitable for settlement. Therefore, numerous archaeological sites with valuable remains dating from the Neolithic to Medieval are known in this area. The site of Jagodnjak-Krčevine represents one of these archaeological settling places located 10 kilometres north-east of the Drava River and 18 kilometres west of the Danube at 91 meters above sea level on the slope of a loess plateau along the Gmajna Stream. The site was excavated in 2014 and 2015 due to the construction of the highway route, revealing the remains of settlements and cemeteries from different periods of the past (Tresić Pavičić, 2015).

The oldest traces of human activities found on the site belong to Early Neolithic communities (6th millennium BC). Discovered pottery (see Figure 1) has the stylistic characteristics of the Starčevo culture i.e. the Starčevo-Körös-Criș cultural group (Balén et al., 2014) and was found inside the remains of a settlement with a system of circular ditches, indicating a permanent settlement. The vessels were hand-made, of simple shape, mainly bowls and pots of thick walls. Most of the archaeological records belong to the Bronze Age, i.e. to the communities of Pannonian Encrusted Pottery (20th – 17th century BC, Kiss, 2012). Bronze Age records consist of a working and resident living area, located on the edge of the settlement. A cemetery with bi-ritual burials was discovered near the settlement. The graves were accompanied by extremely rich grave goods, such as metal jewellery (gold, bronze), metal parts of costumes, and numerous ceramic vessels. The pottery style is very distinctive, especially fine ware with a dark surface decorated with white paste inlay that fills incised motive compositions. The excavation also discovered traces of a settlement complex dated to the Late Iron Age with characteristic dark grey (graphite) pots and wheel-made bowls which present the *La Tène* culture i.e. Celtic pottery tradition

(3rd – 1st century BC). The pottery style corresponds to types and variants of vessel types found in numerous settlements remains of Eastern Slavonia, produced and used by Scordisci communities (**Dizdar, 2001**).

Additionally, the site was occupied shortly after, in the Roman Imperial Period (1st – 4th century AD). The utilitarian ceramics of local production from Jagodnjak correspond to vessel repertoire in the area (**Jelinčić Vučković, 2015; Ožanić Roguljić, 2016**).

This position was again occupied during the Early Middle Ages – with an excavated larger cemetery. Later, during the Medieval (12th – 15th century), a settlement was again built on the site. A small part of the settlement consists of predominantly ceramic material, i.e. simple-shaped pots, most often cooking pots. A similar vessel shape was frequently used during the Middle Ages, and such vessels can be found on archaeological sites in the area of eastern Slavonia and Baranja (**Tkalčec et al., 2017**). According to the excavation results, and the evidence of well-organized communities that chose this location for permanent settling, it seems that the site was an important strategic place, probably chosen because of the good geostrategic features, particularly significant for certain periods of the past (Neolithic, Bronze Age, Late Iron Age, Roman period, Early Middle Ages and Mediaeval). Since the settlements were of a long-term character, and according to the easy availability of raw materials, it is assumed that the pottery production was organized locally.

3. Geological setting

The Baranja region tectonically belongs to the North Croatian Basin as a part of the south-western Pannonian basin system (**Horváth et al., 2006; Pavelić and Kovačić, 2018**). The formation of the Pannonian basin system occurred in the Early Miocene due to the continental collision and subduction of the Adriatic microplate beneath the Pannonian tectonic segment of the Euroasian Plate (**Šumanovac et al., 2017; Pavelić and Kovačić, 2018**). The area is characterized by a long emersion phase that lasted until the Early Miocene. It is a period of continental environments that were replaced by lacustrine and then marine sedimentation during the Middle Miocene (**Kováč et al., 2007; Mandić et al., 2012; Pavelić and Kovačić, 2018**).

According to **Bognar (1990)**, three morphographic types represent the relief of Baranja; lowlands, plains, and hilly relief. The lowland relief accounts for almost 90% of the area and geomorphologically consists mainly of marshes and fluvial lowlands. The most widespread is the fluvial lowland of the Drava River in the central and southwestern part of Baranja, and the Danube River, in the eastern part of this area (**Bognar, 1990**). The formation of the rivers dates back to the Early Pleistocene while a significantly warmer period during the Early Holocene caused the extensive melting of Alpine gla-

ciers and the downstream deposition of clayey, silty, and sandy sediments (**Oreščanin et al., 2005; Lóczy et al., 2014**). The lowland is covered by the Southern and Northern Baranja loess plateau composed of loess and loess-like deposits of aeolian origin (**Bognar, 1990; Galović, 2016**). These well-preserved successions have an excellent preservation potential for traces of climate change and are therefore continuously the subject of numerous studies (**Marković et al., 2009; Galović et al., 2009; 2011; Banak et al., 2012; Galović 2016; Galović and Peh, 2016**). Loess-paleosol successions can reach a thickness of up to 40 m and are commonly intercalated with alluvial deposits composed of loess-derived sediments eroded from the rivers catchments (**Galović, 2016; Galović and Peh, 2016**). During the Late Pleistocene, strong winds formed in a cold glacial climate led to the deposition of loess and loess-like sediments in lakes, ponds, shallow marshes, and on the mild elevations (**Bačani et al., 1999**). Conversely, the formation of paleosols and alluvial sediments indicates warmer periods and fluvial activity (**Galović, 2016**). Different types of loess-like sediments are also prevalent on BANSKO BRDO, the only elevation in the area. Tectonically, it is an asymmetric horst in the NE-SW direction (**Banak et al., 2012**). The oldest exposed rocks belong to the Miocene volcano-sedimentary complex with basalt-andesite and pyroclastic rocks (**Pamić and Pécskay, 1996; Banak et al., 2012**). The formation of this complex is probably related to continental rifting processes during the Pannonian Basin extension (**Horváth, 1995; Pavelić, 2001; Banak et al., 2012; Mandić et al., 2012**).

The multi-period archaeological site Jagodnjak-Krčevine is located in the central Baranja region, near the village of Jagodnjak. In contrast to the very well studied loess-paleosol sequences, there are few investigations explaining the geological background of the study area. Geomorphologically, the most prominent feature in the area is the meandering Drava River with its alluvial terrace. The predominant deposits are represented by sandy and silty sediments, while oxbows and ridges were recorded in the southern part (**Bognar, 1990; Pikić et al., 1991; Pikić et al., 2015**). The river channel migration together with changes in flood level resulted in the formation of oxbows with a predominant deposition of silty, sandy, and sandy clayey sediments (**Pikić et al., 1991**).

4. Sampling and methods

4.1. Field sampling

According to ethnoarchaeological research, the distance between the raw material source and the place of pottery production ranges from 1 to 10 kilometres (**Rice, 1987**). However, raw materials were most often exploited within a radius of 1 km from the settlement where production took place (**Arnold, 2000**). Therefore, field

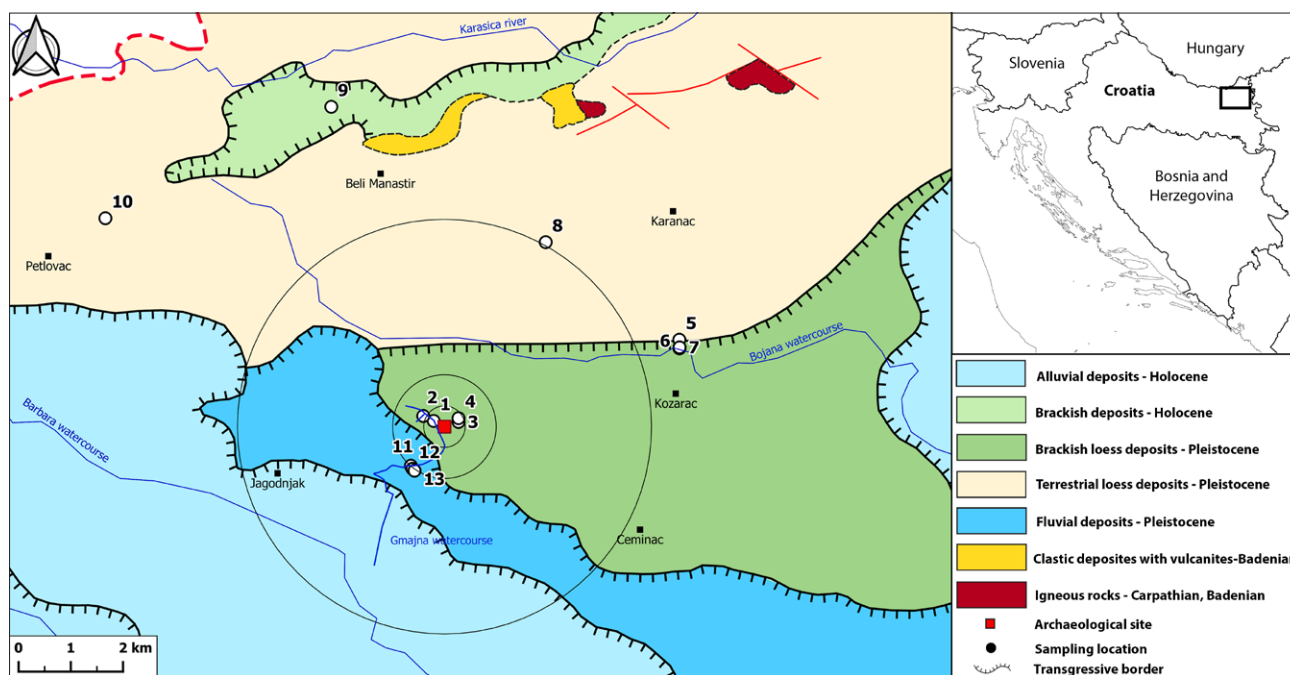


Figure 2: Geological map with sampling locations within a different radius distance from the archaeological site Jagodnjak-Krčevine. Gmajna watercourse – location 1 (samples 9095, 9096, 9097), location 2 (sample 9098), location 11 (sample 9108), location 12 (sample 9109) and location 13 (sample 9110); immediate vicinity of the archaeological site – location 3 (sample 9099) and location 4 (sample 9100); Kozarac old clay pit – location 5 (9101); Bojana watercourse – location 6 (sample 9102) and location 7 (samples 9103, 9104); Karanac old clay pit – location 8 (sample 9105); Beli Manastir old clay pit – location 9 (sample 9106); Petlovac old clay pit – location 10 (sample 9107) (modified after **Croatian Geological Survey, 2009**).

sampling was concentrated in the vicinity of the archaeological site, near the present small watercourses, which are a potentially good location for collecting fine-grained clay sediments. Several locations of recently exploited clay pits (**Pikija et al., 1991**) in the study area were chosen as the primary sampling sites. Samples were collected in several zones: near the archaeological site (400-meter radius from the archaeological site), and in zones within 1, 4, and 10 km radius (see **Figure 2**). During the fieldwork, samples of clay sediments were collected using a hand auger from 0.3 and max 1.7 m depth. A total of 16 samples were collected from 13 locations and subsequently prepared for physical (plasticity and shrinkage), granulometric, mineralogical, and petrographic analyses. Plasticity was measured on-site, by simulating actual processing conditions and by manual modelling a clay roller and observing deformation due to bending force (plasticity range: no plasticity, low, moderate, and high plasticity).

4.2. Analytical Methods

Two types of samples were analysed: archaeological and geological. Archaeological samples include 31 ceramic sherds dated to five different periods. The archaeological data and main physical characteristics of sherds are presented in **Table 1**. Geological samples are represented by 16 samples of clayey sediments collected in the vicinity of the archaeological site. Analyses present in this study were conducted at the University of Zagreb,

Faculty of Mining, Geology and Petroleum Engineering and at the Institute of Archaeology in Zagreb.

4.2.1. Clay briquettes and test rollers - firing and shrinkage

In order to examine the clayey material consistency and microtexture after firing, and enable correlation with archaeological ceramic sherds, experimental clay briquettes were made from the collected clayey materials, i.e. from the samples with moderate and high plasticity observed on site (12 out of 16 samples). By simulating the actual processing conditions, the samples were freed from coarser fractions and plant inclusions, and each was kneaded until it reached the optimal workable condition. Despite the same storage conditions, a very wide range of processability was found for the different samples. Few samples were already in a plastic state that allowed the clay to be modeled – mostly without the presence of coarse mineral inclusions or plant remains that had to be removed. Extremely dry samples, where it was not possible to achieve optimal workability, were soaked in water for several days and after removing the excess water, the samples were dried until they became workable. The samples were molded in 4 x 6 x 1.5 cm briquettes and dried at room temperature for several days. They were fired in a laboratory kiln at 650°C under controlled conditions for one hour with a 30-minute soaking period (holding the kiln at final firing temperature) and then allowed to cool overnight. Short firing

Table 1: Archaeological data and main physical characteristics of the vessels from the Jagodnjak-Krčevine site (PEP – Pannonian Encrusted pottery, SM – smoothed, UN – untreated, ROU – roughed)

Sample number	Archaeol. Context	Vessel type	Period	Style / Culture	Manufacturing technique	Wall thickness (cm)	External Surface Treatment	Inner Surface Treatment	Firing methods
8987	Settlement	unknown	Neolithic	Starčevo	Coiling / Slab tech.	0.9	SM	SM	Open fire
8988	Settlement	pot	Neolithic	Starčevo	Coiling / Slab tech.	1	SM	SM	Open fire
8989	Settlement	bowl	Neolithic	Starčevo	Coiling / Slab tech.	1	UN	SM	Open fire
8990	Settlement	pot	Neolithic	Starčevo	Coiling / Slab tech.	0.5	SM	SM	Open fire
8992	Settlement	pot	Neolithic	Starčevo	Coiling / Slab tech.	0.5	SM	SM	Open fire
8998	Settlement	pot	Neolithic	Starčevo	Coiling / Slab tech.	0.5	SM	SM	Open fire
9456	Grave 056	fine pot	Bronze Age	PEP	Coiling / Slab tech.	0.6	SM	SM	Open fire/Kiln
9432	Grave 060	fine pot	Bronze Age	PEP	Coiling / Slab tech.	0.4	SM/ROU	SM	Open fire/Kiln
9457	Grave 056	fine pot	Bronze Age	PEP	Coiling / Slab tech.	0.5	SM/ROU	SM	Open fire/Kiln
9458	Grave 060	fine pot	Bronze Age	PEP	Coiling / Slab tech.	0.6	SM/ROU	SM	Open fire/Kiln
9459	Grave 059	fine pot	Bronze Age	PEP	Coiling / Slab tech.	0.4	SM/ROU	SM	Open fire/Kiln
9433	Grave 056	fine pot	Bronze Age	PEP	Coiling / Slab tech.	0.3	SM/ROU	SM	Open fire/Kiln
9460	Grave 002	coarse pot	Bronze Age	PEP	Coiling / Slab tech. / Beating	0.6	SM/ROU	SM	Open fire
9461	Grave 006	coarse pot	Bronze Age	PEP	Coiling / Slab tech. / Beating	0.8	SM/ROU	SM	Open fire
9462	Grave 059	coarse pot	Bronze Age	PEP	Coiling / Slab tech. / Beating	0.6	SM/ROU	SM	Open fire
9434	Grave 027	coarse pot	Bronze Age	PEP	Coiling / Slab tech. / Beating	0.7	SM/ROU	SM	Open fire
9435	Grave 061	coarse pot	Bronze Age	PEP	Coiling / Slab tech. / Beating	1.0	SM/ROU	SM	Open fire
8991	Settlement	graphite pot	Late Iron Age	La Tène	Coiling / Slab tech.	0.6	SM	UN	Kiln
8995	Settlement	graphite pot	Late Iron Age	La Tène	Coiling / Slab tech.	0.6	SM	SM	Kiln
9000	Settlement	fine bowl	Late Iron Age	La Tène	Wheel-thrown	0.5	SM	SM	Kiln
8753	Settlement	graphite pot	Late Iron Age	La Tène	Coiling / Slab tech.	0.8	SM	SM	Kiln
8986	Settlement	lid	Roman period	Roman	Wheel-coiling	0.5	SM	SM	Kiln
8993	Settlement	pot	Roman period	Roman	Wheel-coiling	0.6	SM	SM	Kiln
8994	Settlement	pot	Roman period	Roman	Wheel-coiling	0.3	UN	UN	Kiln
8997	Settlement	pot	Roman period	Roman	Wheel-coiling	0.4	SM	SM	Kiln
9001	Settlement	bowl	Roman period	Roman	Wheel-thrown	0.6	SM	UN	Kiln
8996	Settlement	pot	Medieval	Medieval	Wheel-coiling	0.5	SM	SM	Open fire/Kiln
8999	Settlement	pot	Medieval	Medieval	Wheel-coiling	0.6	SM	SM	Open fire/Kiln
9002	Settlement	pot	Medieval	Medieval	Wheel-coiling	0.7	SM	SM	Open fire/Kiln
9003	Settlement	pot	Medieval	Medieval	Wheel-coiling	0.5	SM	UN	Open fire/Kiln
8752	Settlement	pot	Medieval	Medieval	Wheel-coiling	0.7	SM	SM	Open fire/Kiln

time and low temperatures are assumed for most archaeological ceramics, especially for prehistoric pottery. Therefore, the aim was to mimic prehistoric firing conditions to obtain conditions for more relevant comparisons of briquettes and archaeological ceramics. The briquettes were prepared without the addition of temper to reveal the differences in composition of the potential raw materials and, if possible, enable the correlation with ceramic material matrix. In addition, the remaining parts of these samples were used for examination of the physical property (shrinkage). 50 g of each sample was modelled in a roller of 7 x 2.5 cm size. The length and diameter of each roller was measured after 24, 48, 72, and 96 hours, re-

spectively, until they were dry. Relative shrinkage was assessed by the difference between the original and dried volumes of each sample.

4.2.2. Grain size analysis

Grain size analysis was conducted on 16 sediments collected on-site. Before analyzing, samples were soaked in distilled water for several hours and then put in an ultrasonic bath to achieve better disintegration. Grain size analysis was conducted using Malvern Panalytical Mastersizer 3000 Particle Size Analyzer with Hydro EV unit and < 5% obscuration.

4.2.3. Petrography

Thin sections of the pottery sherds (31 samples) and the fired clay briquettes (12 samples) were examined under a polarizing microscope Zeiss Axiolab 5 in transmitted light. The petrographic analysis of ceramics includes the determination of the colour of the matrix, its texture, matrix birefringence, mineral composition, the presence of natural and anthropogenic inclusions, voids, and pores. During the macroscopic description and the petrographic analysis, the inclusion density, size, sorting, and roundness of the grains were determined according to the instructions of the Prehistoric Ceramic Research Group (PCRG, 2010). Petrography of ceramics enables determination of ceramic fabric groups which indicate the compositional differences among the clay paste. The definition of tempering material is based on the type, size, shape, and bimodal grain size distribution in the matrix.

4.2.4. Mineralogical analyses

Mineralogical analysis by XRD was performed on ceramics (24 samples from all the determined fabric groups and ceramics from all studied periods), as well as clayey material samples (16 samples). Bulk samples (of both types) have been crushed to a powder fraction using a Retsch vibratory disc mill RS 200. In order to better define clay minerals within clayey material, <2 micrometers fraction, as well as oriented films of the separated clay fraction were subjected to various treatments (glycolated, dimethyl sulfoxide intercalated, heated 550°C) and analysed. A clay fraction was analysed on samples with “moderate” and “high” plasticity observed on site. Interpretation of clay mineral content was done according to **Moore and Reynolds (1997)**.

The mineralogical composition was determined using a Panalytical Empyrean diffractometer equipped with a graphite monochromator and Cu-K α radiation. Scan settings were 3–70 °2 θ on the rotating sample stage, 0.013 °2 θ step size, 1 s count time per step. Mineral identification of samples was conducted using PANalytical X'Pert HighScore software with standardized Powder Diffraction Files (PDF) of the International Centre for Diffraction Data (ICDD) (NewtonSquare, PA, USA).

5. Results

For better understanding, the overall results of this study are divided into two categories: archaeological and geological samples. The results of each analysis conducted on both ceramic and clayey samples are presented below.

5.1. Archaeological ceramic

5.1.1. Petrography

The mineral composition of ceramic clay matrix determined by optical microscopy is mainly composed of

very fine (< 0.1 mm) quartz and mica crystalloclast inclusions (see **Table 2**). K-feldspar is identified in most of the samples while plagioclase is less common. Mineral phases such as biotite, pyroxene, and zircon are very rarely present. Larger quartz grains sometimes contain cracks. By observing the density and size of mineral inclusions in the matrix, ten main fabric groups have been determined (see **Table 2, Figure 3**). In general, the density of very fine crystalloclasts varies from moderate (15%) to abundant (50%) while only one sample stands out with less than 5% (8994) of crystalloclast inclusions. Mineral inclusions are well to very well distributed in the matrix in all samples. Besides very fine crystalloclast inclusion, some samples also contain fine (0.1 – 0.25 mm) crystalloclasts whose density varies from rare (2%) to moderate (15%).

According to the mineral composition of the clay matrix, two general groups of raw materials were observed. The first one is dominated by quartz, while the second is dominated by mica. Quartz-rich matrix is determined in seven fabric groups, representing 80% of all samples and characterized by angular-subangular mono-crystalline quartz grains. The matrix colour varies from orange to dark brown while the birefringence is low to high but mainly medium high. Micaceous matrix is less common, determined in three fabric groups, and therefore present in 20% of all samples. It is dominated by a large number of mainly elongated very fine to fine mica minerals. The colour of the matrix is light brown to brown with the birefringence varying from medium-high to high.

Along with crystalloclasts, rock fragments (lithoclasts) are present in 12 samples (39% of total samples). They are mainly represented by metamorphic rocks, quartzite, and less common schist. Among sedimentary rocks, the most common is limestone while sandstones and chert are present in smaller amounts. Igneous rocks are rarely present. The characteristic lithoclasts are sub-rounded and sub-angular, fine to coarse in size (0.1–3 mm) with moderate to very common amounts (10 – 30%). Lithoclasts are generally poorly to moderately sorted in the matrix, although there are several samples with well sorted grains. The amount and bimodal grain size distribution suggest that medium and coarse lithoclasts were added to the clay intentionally as temper.

Moreover, grog (ceramoclasts) is determined in seven samples (23%). Grog grains are sub-angular to sub-rounded with the majority having sub-rounded shapes. The grog size varies from medium to coarse and is medium to poorly sorted in the matrix. The composition of grog grains is usually very similar to the composition of the ceramic matrix and the main difference is in the colour and sometimes in the amount of very fine crystalloclasts. Clay pellets/ARF (argillaceous rock fragments) were recorded in negligible quantity. The majority of them are transparent, with some being opaque, having sharp, clear, or diffuse edges. The amount and size of the pores are variable from sample to sample but in general,

they vary from 2% to 15% (average: 6.3 %). Three samples or 10% of all analysed samples have no visible pores (less than 1%). They are mostly elongated, parallel to the vessel walls while rounded and irregular pores are present but less common. In five samples, organic material is recorded in mostly elongated and curved voids. Moreover, fragments of shells, medium to coarse in size are recorded in four samples in mostly elongated and slightly curved shapes. Fragments are present in sparse amounts (<5%) and are therefore probably naturally present, with clearly visible inner structure. By observing the type, size, shape, and bimodal grain size distribution in the clay matrix 19 fabric subgroups have been determined (see **Table 2**).

Fabric 1.1: The main naturally present crystalloclast inclusions are very fine (< 0.1 mm) and fine (0.1 – 0.25 mm) monocrystalline quartz with the occurrence of mica minerals. Grains are sub-angular and very well distributed in the clay. The amount of very fine crystalloclasts in the matrix is moderate to common (15 – 20%) while the density of fine crystalloclasts is sparse to moderate (5 – 10%). Moreover, slightly curved, and mostly elongated shell fragments are present in sparse amounts (<5%).

Fabric 1.2: The main characteristic of this subgroup is grog tempering. This fabric contains between 7 and 15% of grog. The grains are medium to coarse (0.25 – 3 mm), sub-angular to sub-rounded, and generally moderately sorted with one sample showing well sorted grains. Grog has a similar mineral composition and brownish colour as a clay matrix with sporadically vitrified grains. The main naturally present crystalloclast inclusions are very fine and fine monocrystalline quartz with the occurrence of mica minerals and the rare presence of K-feldspar. Grains are sub-angular and very well distributed in the clay. The characteristics of naturally present crystalloclast inclusions correspond to the subgroup 1.1.

Fabric 1.3: Grog and lithoclast tempering is characteristic for this group. This fabric contains around 15% of grog and 10% of lithoclasts. Grog grains are medium in size, sub-rounded, and moderately sorted in the clay. Grog grains contain slightly greater amount of fine crystalloclast inclusions than the matrix and are darker in colour. Lithoclasts are composed of metamorphic rocks, mainly quartzite. These are fine to medium in size, sub-angular and sub-rounded, moderately sorted in the clay. The characteristics of naturally present crystalloclast inclusions correspond to the subgroup 1.1.

Fabric 2.1: This subgroup is characterized by lithoclast tempering. Lithoclasts are composed of metamorphic rocks, mostly quartzite with the sporadic occurrence of igneous rocks. The grains are sub-angular, present in a common amount (25%), and poorly sorted. Naturally present crystalloclast inclusions are composed of very fine monocrystalline quartz and mica minerals. The density of very fine crystalloclasts in the matrix is common (20%) and the grains are sub-angular and very well distributed in the clay.

Fabric 2.2: Unimodal grain size distribution in this subgroup suggests no added tempering material. However, elongated shell fragments with visible inner structure are present in sparse amounts (<5%). The characteristics of the clay matrix are the same as in subgroup 2.1.

Fabric 2.3: This subgroup is characterized by grog and lithoclast tempering. Grog is present in sparse amounts (5%) and the grains are coarse, sub-rounded, and moderately sorted in the matrix. When compared with the clay matrix, grog grains possess similar mineral composition and are slightly darker colour. Lithoclasts are composed of metamorphic (quartzite) and less common sedimentary rocks (sandstone). The grains are fine to medium in size, moderately sorted with sub-angular and sub-rounded shapes. Sandstone inclusions seem to be more rounded. Naturally present crystalloclast inclusions are very fine monocrystalline quartz and mica minerals with the occurrence of K-feldspar. The characteristics of the clay matrix inclusions are the same as in subgroup 2.1.

Fabric 3.1: The main characteristic of this subgroup is the presence of elongated and curved voids in amounts between 10 and 15%. It is assumed that the voids retained the form of plant material added to the paste before firing. In several samples, black carbonized remains of plant inclusions occur within curved voids. The main naturally present crystalloclast inclusions are very fine and fine monocrystalline quartz with the occurrence of mica minerals and the rare presence of K-feldspar. Grains are sub-angular and very well distributed in the clay. The amount of very fine crystalloclasts in the matrix is abundant (40%) with sparse occurrence (5%) of fine crystalloclast inclusions.

Fabric 3.2: Regarding the absence of medium and coarse inclusions in the matrix, this fabric subgroup has no tempering material. The main naturally present crystalloclast inclusions in this subgroup are very fine and fine monocrystalline quartz with the occurrence of mica minerals and K-feldspar. Plagioclase, biotite, and zircon are determined in some samples. The density of naturally present crystalloclast inclusions in the matrix corresponds to the subgroup 3.1.

Fabric 3.3: This subgroup is characterized by grog tempering in the amount between 7 and 10 %. The grains are medium to coarse, sub-rounded, and moderately sorted. Grog grains tend to have the same colour and mineral composition as the matrix and are therefore sometimes hard to recognize. The characteristics and mineral composition of clay matrix correspond to the subgroup 3.1.

Fabric 4.1: This group is characterized by both lithoclasts and graphite tempering. Prevailing lithoclasts are metamorphic (quartzite) with a rare occurrence of sedimentary rocks (chert). The density of lithoclasts is moderate (15%), grains are medium to coarse, sub-angular and moderately sorted in the matrix. The significant characteristic of this group is the presence of opaque

Table 2: Ceramic petrography analysis of pottery sherds from Jagodnjak-Krčevine site (L – low, MH – medium high, H – high; F – fine, M – medium, C – coarse; PS – poorly sorted, MS – medium sorted, WS – well sorted, SA – sub-angular, SR – sub-rounded; OM – organic material, ** – plant material, * – shell fragments)

Sample number	Birefringence	Very fine crystalloclasts	Fine crystalloclasts	Quartz	K-feldspar	Plagioclase	Muscovite	Biotite	Pyroxene	Zircon	Lithoclasts density	Lithoclasts size	Lithoclasts sorting	Lithoclasts shape	Metamorphic	Igneous	Sedimentary	Graphite	Grog density	Grog size	Grog sorting	Grog shape	MO	Fabric groups	Type of raw material
8999	MH			+		+																	*	1.1	Quartz-rich
9435	MH			+	+	+													15%	M/C	MS	SR		1.2	
9457	MH	15-20%	5-10%	+	+	+													7%	M	MS	SR		1.2	
9461	MH			+	+	+													15%	M/C	MS	SR		1.2	
9432	H			+	+	+					10%	F/M	MS	SA/SR	+				15%	M	MS	SR		1.3	
8993	H			+		+	+				25%	F/C	PS	SA	+	+								2.1	
8997	H	15-20%		+	+	+																	*	2.2	
9460	H			+	+	+					10%	F/M	MS	SA/SR	+		+	5%	C		MS	SR		2.3	
8987	MH			+		+																	**	3.1	
8988	MH			+	+	+																	**	3.1	
8998	MH			+		+																	**	3.1	
9000	MH			+	+	+																		3.2	
9456	MH	40-50%		+	+	+																		3.2	
9458	MH		5%	+	+	+																		3.2	
9459	H			+		+		+	+	+														3.2	
9433	MH			+	+	+		+																3.2	
9462	MH			+	+	+													7%	M/C	MS	SR		3.3	
9434	H			+	+	+													10%	M/C	MS	SR		3.3	
8991	L	20-25%	10%	+	+	+	+	+	+	+	15%	M/C	MS	SA	+		+	+						4.1	
8753	L			+	+	+		+	+	+	15%	M/C	MS	SA	+		+	+						4.1	
8994	MH	<5%		+		+		+			30%	F/C	PS	SA	+	+								5.1	
8989	MH			+	+	+																		6.1	
8990	H	35-40%		+	+	+																	**	6.2	
8995	L	25%		+	+	+					15%	F/C	MS	SA	+	+	+	+						7.1	
9003	MH			+	+	+					30%	M/C	WS	SA	+									7.2	
8996	H			+	+	+					20%	M/C	MS	SA	+	+	+							8.1	
9002	MH	25%		+	+	+					25%	M/C	PS	SA	+	+	+						*	8.1	
8752	H			+	+	+					20%	M/C	MS	SA	+								*	8.1	
8986	H			+	+	+		+																9.1	
9001	H	40%		+	+	+		+					WS	SA	+	+	+							9.2	
8992	MH	40%	5%	+	+	+					30%	M											**	10.1	

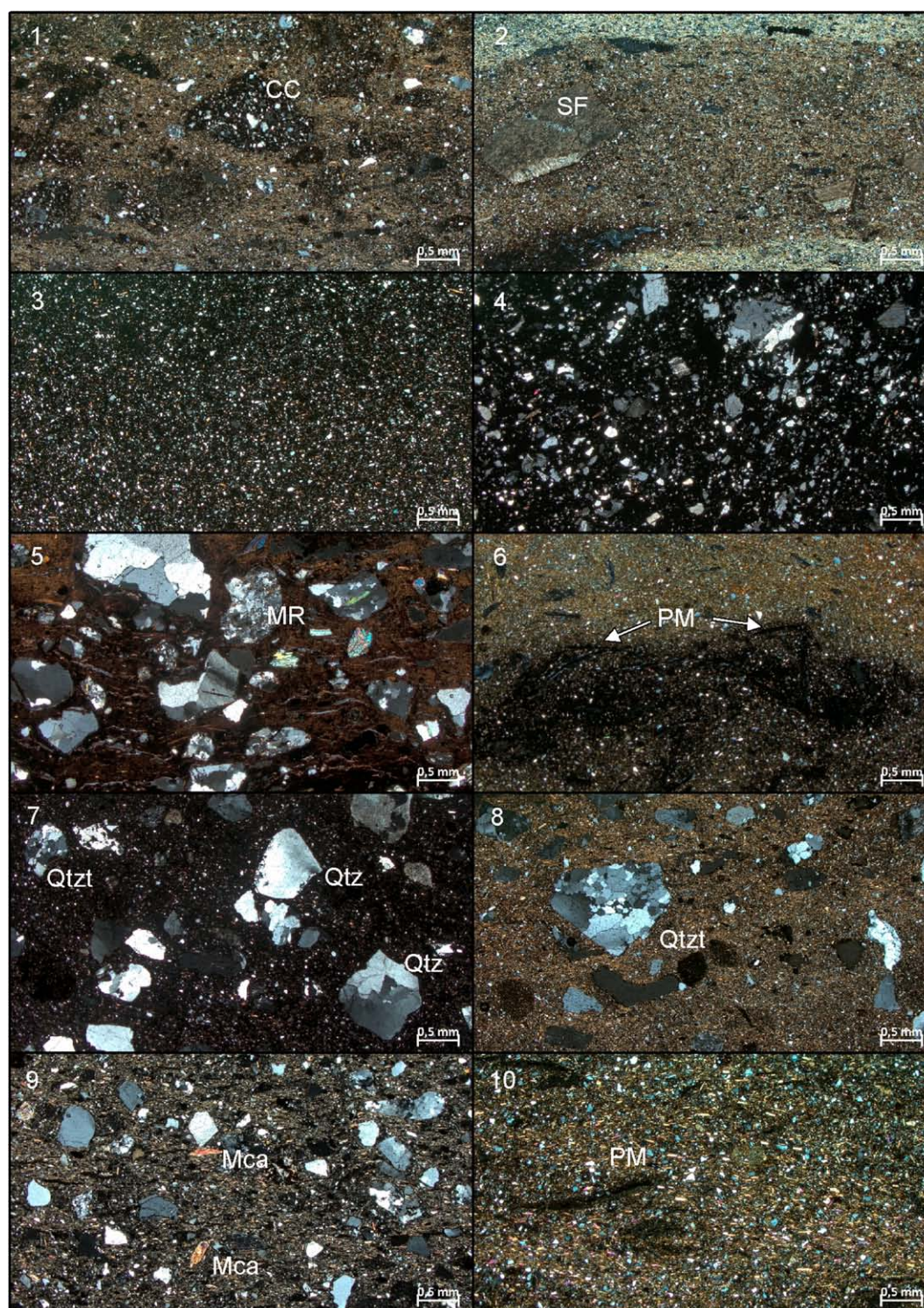


Figure 3: Thin section microphotographs of different fabric groups according to the matrix microtexture (XP).

Fabric group 1 – sample 9432; Fabric group 2 – sample 8997; Fabric group 3 – sample 9000;
 Fabric group 4 – sample 8991; Fabric group 5 – sample 8994; Fabric group 6 – sample 8990;
 Fabric group 7 – sample 9003; Fabric group 8 – sample 8752; Fabric group 9 – sample 9001;
 Fabric group 10 – sample 8992; (Mca – mica, Qtz – quartz, Qtzt – quartzite, MR – metamorphic rock,
 SF – shell fragment, PM – plant material, CC – ceramoclast/grog).

graphite grains which cause the extremely dark matrix colour and low birefringence. These grains are present in very common amounts (30%), fine to coarse in size and angular to subangular in shape. The main naturally pre-

sent crystalloclast inclusions are composed of very fine and fine monocrystalline quartz. Mica minerals, K-feldspar, and plagioclase are also present in the samples. The density of very fine crystalloclasts in the matrix is com-

mon (20%) and the grains are sub-angular and very well distributed in the clay. Fine crystalloclast inclusions are present in moderate (10%) amounts.

Fabric 5.1: This group is also characterized by lithoclast tempering. Lithoclasts are very common (30%), composed of metamorphic (quartzite and less common schist) and igneous rocks. They are mainly sub-angular, fine to coarse in size and poorly sorted in the matrix. Naturally present crystalloclast inclusions are composed of very fine monocrystalline quartz and mica minerals present in sparse amounts (<5%). These are sub-angular and very well distributed in the clay.

Fabric 6.1: According to unimodal grain size distribution, this subgroup has no added temper material in the paste. Naturally present crystalloclast inclusions are composed of very fine monocrystalline quartz and less common K-feldspar and mica minerals present in very common amounts (35%). Grains are sub-angular and well distributed in the clay.

Fabric 6.2: This subgroup is characterized by the presence of elongated and curved voids in amount around 15% that retained the form of plant material added to the paste before firing. As in the subgroup 3.1, sometimes black carbonized remains of plant occur within curved voids. The characteristics and mineral composition of clay matrix correspond to the subgroup 6.2.

Fabric 7.1: Like in Fabric 4.1, the characteristics of this subgroup are both lithoclast and graphite tempering. Characteristic lithoclasts are metamorphic rocks, mostly quartzite and less common schist while igneous rocks are rarely present. These grains are sub-angular, present in moderate amounts (15%) and moderately sorted in the matrix. The graphite grains are opaque, present in common amount (20%), with sub-angular shape and medium to coarse in size. Naturally present crystalloclast inclusions are composed of very fine monocrystalline quartz with the occurrence of K-feldspar and mica minerals. These are sub-angular, well distributed and common in the clay (25%).

Fabric 7.2: The main characteristic of this subgroup is lithoclast tempering. Characteristic lithoclasts are metamorphic rocks, mostly quartzite and less common schist while igneous rocks are rarely present. Grains are sub-angular, present in common amounts (30%) and are well sorted in the matrix. The characteristics and mineral composition of clay matrix correspond to the subgroup 7.1.

Fabric 8.1: Similar to previous one, this group is also characterized by lithoclast tempering. Among lithoclasts, all three rock types are determined in the samples but metamorphic lithoclasts prevail (quartzite). Sedimentary lithoclasts are mostly determined as limestone. The grains are medium to coarse, sub-angular and poorly to moderately sorted in the matrix. The lithoclast density varies between 20 and 25%. The main naturally present crystalloclast inclusions are very fine elongated mica minerals with the occurrence of quartz, K-feldspar and plagioclase minerals. Grains are sub-angular and well distributed in the clay with contents up to 25%.

Fabric 9.1: The main naturally present crystalloclast inclusions are composed of very fine elongated mica minerals with the common presence of quartz and K-feldspar. Biotite and plagioclase are rarely present. Grains are sub-angular and well distributed in the clay. Very fine crystalloclast inclusions are present in abundant (40%) amounts. The only coarse inclusions recorded in this subgroup are slightly curved and elongated shell fragments present in sparse amounts (<5%) and probably naturally present.

Fabric 9.2: The main characteristic of this group is lithoclast tempering. Lithoclasts are mostly composed of metamorphic rocks (quartzite and schist) while sedimentary rocks (limestone) are present in smaller amounts. Lithoclasts are very common (30%), sub-angular, medium in size and well sorted in the matrix. The characteristics and mineral composition of clay matrix correspond to the subgroup 9.1.

Fabric 10.1: This group is characterized by elongated and curved voids present in sparse amounts (7%). The occurrence of black carbonized remains of plant material within curved voids is very common. The main natu-

Table 3: Qualitative mineralogical composition of pottery samples determined by X-ray diffraction analysis (Qtz – quartz, Pl – plagioclase, Kfs – K-feldspar, Mca – micaceous minerals, Cal – calcite, Dol – dolomite, Amph – amphibole, Grph – graphite, Hem – hematite, Crs – cristobalite, + – present, n.d. – not detected)

Sample number	Qtz	Pl	Kfs	Mca	Cal	Dol	Amph	Grph	Hem	Crs
8986	+	+	+	+	+	+	+	n.d.	n.d.	n.d.
8989	+	+	+	+	+	+	+	n.d.	n.d.	n.d.
8994	+	+	+	+	n.d.	n.d.	+	n.d.	n.d.	n.d.
8995	+	+	+	+	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
8997	+	+	+	+	+	n.d.	+	n.d.	n.d.	n.d.
8998	+	+	n.d.	+	n.d.	n.d.	+	n.d.	n.d.	n.d.
8999	+	+	+	+	+	n.d.	n.d.	n.d.	n.d.	n.d.
9000	+	+	+	+	n.d.	n.d.	+	n.d.	+	n.d.
9001	+	+	+	+	+	n.d.	n.d.	n.d.	n.d.	n.d.
9002	+	+	+	+	+	+	n.d.	n.d.	n.d.	n.d.
9003	+	+	+	+	+	n.d.	+	n.d.	n.d.	n.d.
8752	+	+	+	+	+	n.d.	n.d.	n.d.	n.d.	n.d.
8753	+	+	+	+	n.d.	n.d.	n.d.	+	n.d.	n.d.
9432	+	+	+	+	n.d.	n.d.	+	n.d.	n.d.	n.d.
9433	+	+	+	+	+	n.d.	+	n.d.	n.d.	n.d.
9434	+	+	+	+	n.d.	n.d.	+	n.d.	n.d.	+
9435	+	+	+	+	+	n.d.	+	n.d.	n.d.	+
9456	+	+	+	+	n.d.	n.d.	+	n.d.	n.d.	n.d.
9457	+	+	+	+	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
9458	+	+	+	+	n.d.	n.d.	+	n.d.	n.d.	n.d.
9459	+	+	+	+	n.d.	n.d.	+	n.d.	n.d.	n.d.
9460	+	+	+	+	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
9461	+	+	+	+	+	n.d.	+	n.d.	n.d.	n.d.
9462	+	+	+	+	+	n.d.	n.d.	n.d.	n.d.	n.d.

rally present crystalloclast inclusions are composed of very fine elongated mica minerals with quartz and K-feldspar occurrence. The amount of very fine crystalloclasts in the matrix is very common (40%), the grains are sub-angular and very well distributed in the clay. Besides that, the subgroup contains fine crystalloclast inclusions present in sparse amounts (5%).

5.1.2. Mineralogy

The mineralogical composition of the archaeological ceramic determined by XRD generally confirmed the optical microscopy observations (**Table 3**). The presence of quartz and plagioclase is characteristic for all samples while K-feldspar and amphiboles are practically ubiquitous. Calcite is frequently documented and may be accompanied by a sporadic occurrence of dolomite, which are assigned here as primary phases since no secondary calcite was detected by optical microscopy. The common feature of all the analysed samples is the distinct reflex at 10Å interpreted as the first basal peak of the micaceous phyllosilicate. Micaceous material determined by XRD analysis is in accordance with optical microscopy results, classified as muscovite and biotite. Graphite and hematite are recorded in only one sample (8753 and 9000, respectively), while cristobalite was detected in two samples (9434 and 9435).

5.2. Clayey sediments

5.2.1. Physical properties

According to the macroscopic description observed while sampling, most of the collected samples were clas-

sified as clayey sediments with brown to grey colour (see **Table 4**). Some of them, especially those collected near the Gmajna watercourse, contain shell fragments whereas those from old clay pits generally did not have any coarse inclusions. Despite generally similar macroscopic descriptions, they possess significantly different plasticity ranges according to the initial examination observed. Among all of the studied samples, those with “high plasticity” were collected in the immediate vicinity of the archaeological site (Gmajna watercourse) and in an abandoned Petlovac clay pit. The plasticity range of other samples varies from “low” to “moderate” while “no plasticity” was observed in three samples (see **Table 4**). The relative shrinkage of samples varies from approximately 6 to 33% (see **Table 4**). The high plasticity samples generally possess greater shrinkage values (>20%). The samples with the lowest shrinkage values are those collected near the Gmajna (sample 9097 and 9098) and Bojana watercourse (sample 9103).

5.2.2. Grain size analysis

The grain size distribution analysis suggests three different sediment types (see **Figure 4**). The majority of the samples contain more than 75% of silty fraction and less than 23% of clay fraction and are therefore classified as silt. Four samples with a lower silt proportion (65 – 74%) and a greater amount of clay (25% – 33%) are determined as clayey silt. Besides that, only two samples stand out showing a significantly different grain size distribution. They are characterized by a higher amount of sandy fraction (around 50%) and a very low clay con-

Table 4: List of potential raw materials with sampling location and depth, plasticity range, colour (according to the Munsell colour chart), coarse inclusions and shrinkage (/ - not measured). *The clay samples were collected from the exploitation pits where the actual depth was generally greater than 4m.

Sample number	Sampling location	Depth (cm)	Plasticity	Munsell colour	Colour	Coarser inclusions	Shrinkage (%)
9095	1	60-70	high	10YR 2/1	black	shells	28
9096	1	90-100	high	2.5Y 2.5/1	dark grey	shells	33
9097	1	110-120	moderate	2.5Y 4/1	black	shells	10
9098	2	80-90	high	2.5Y 6/4	light yellowish brown	shells	6
9099	3	160-170	no plasticity	5Y 5/2	olive grey		/
9100	4	90-100	moderate	10YR 5/4	yellowish brown		19
9101	5	20-30	no plasticity	2.5Y 5/4	Light olive brown		/
9102	6	*	moderate	2.5Y 4/2	dark greyish brown		17
9103	7	70-80	moderate	2.5Y 4/1	dark grey		9
9104	7	40-50	moderate	2.5Y 3/1	very dark grey		17
9105	8	30-40	moderate	2.5Y 4/4	olive brown		16
9106	9	70-80	moderate	2.5Y 4/2	dark greyish brown		21
9107	10	*	high	2.5Y 5/2	greyish brown		37
9108	11	90-100	high	2.5Y 4/2	dark greyish brown	shells	17
9109	12	*	no plasticity	5Y 4/1	dark grey		/
9110	13	40-50	low	10YR 4/3	brown		/

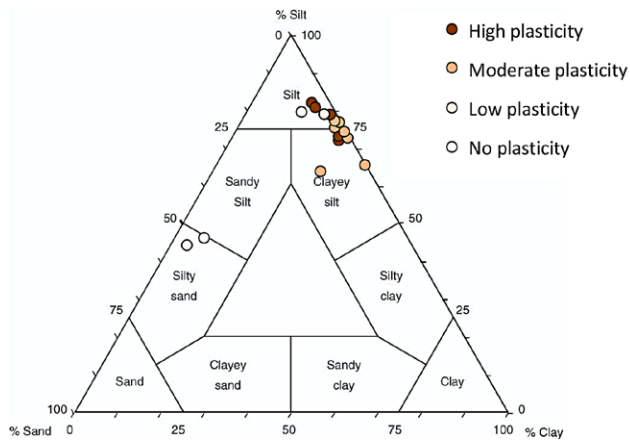


Figure 4: Grain size distribution of clayey sediments collected in the vicinity of the archaeological site correlated with the plasticity range measured on-site

tent, ranging from 4% to 9%, and are therefore classified as silty sands.

5.2.3. Petrography

The mineral composition of fired clay briquettes is dominantly composed of angular-subangular mono-crystalline quartz inclusions with the sporadic occurrence of mica (see **Table 5**). K-feldspar and plagioclase are rarely noticeable. By observing the density and size of mineral inclusions in the matrix of fired clay briquettes, nine fabric groups have been determined (see **Figure 5**). The size and density of inclusions vary among samples. Very fine crystalloclasts inclusions are present

in all the studied samples with density varying from moderate (15%) to abundant (40%). The majority of samples also contain fine crystalloclasts inclusions but in a lower amount, generally up to 7%. Nevertheless, two samples stand out possessing a significantly higher (15 – 20%) amount of fine crystalloclasts inclusions (9106 and 9108). Optical microscopy revealed the presence of shell fragments in eight samples collected along the Gmajna and Bojana watercourse (sampling locations

Table 5: Petrography analysis of potential raw materials collected near the archaeological site (L – low, MH – medium high; + – present)

Sample number	Birefringence	Very fine crystalloclasts	Fine crystalloclasts	Quartz	Feldspar	Muscovite	Biotite	Shells	Fabric group
9095	L	20%	5%	+	+	+		+	1
9097	L			+	+	+		+	
9096	L	15%	3%	+		+		+	2
9098	MH			+	+	+			
9100	MH	35%	5%	+	+	+			3
9102	MH	15%	7%	+	+	+		+	4
9103	MH	30%	5%	+	+	+		+	5
9104	MH			+	+	+		+	
9105	MH	40%		+	+	+			6
9106	MH	30%	15%	+	+	+	+	+	7
9107	MH	25%		+	+	+			8
9108	MH	20%	15%	+	+	+	+	+	9

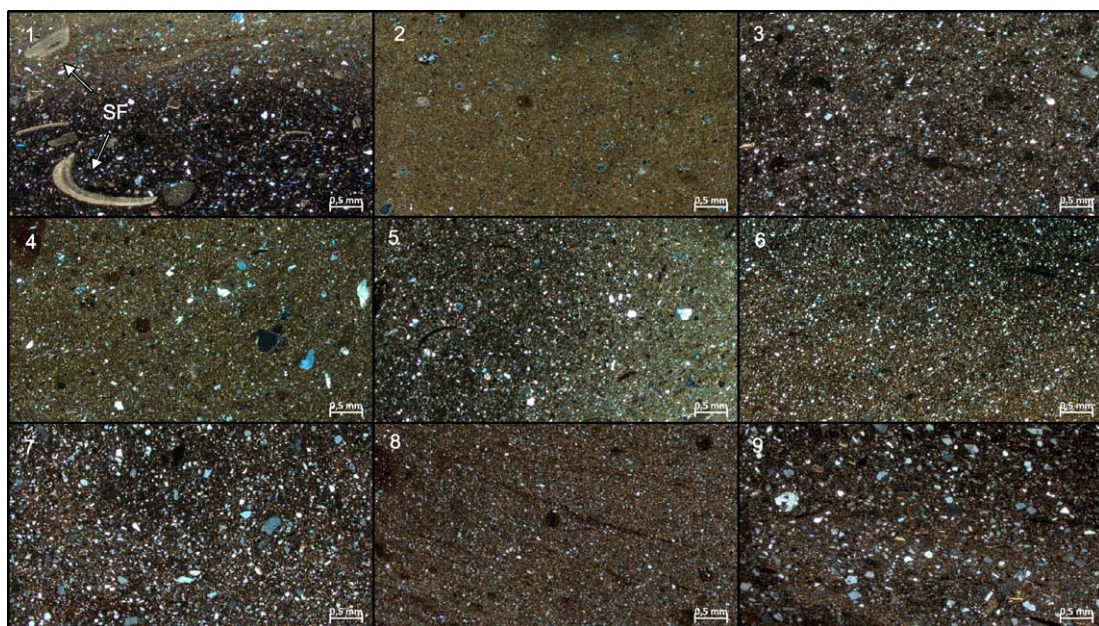


Figure 5: Microphotographs of nine fabric groups of fired clay briquettes determined according to matrix microtexture (XP). Fabric group 1 – samples 9095 and 9097; Fabric group 2 – samples 9096 and 9098; Fabric group 3 – sample 9100; Fabric group 4 – sample 9102; Fabric group 5 – samples 9103 and 9104; Fabric group 6 – sample 9105; Fabric group 7 – sample 9106; Fabric group 8 – sample 9107; Fabric group 9 – sample 9108 (SF – shell fragment).

Table 6: Semi-quantitative mineralogical composition of clayey samples determined by X-ray diffraction analysis (Qtz – quartz, Pl – plagioclase, Kfs – K-feldspar, Mca – micaceous material; including mica, illite and illite/smectite, Cal – calcite, Dol – dolomite, Arg – aragonite, Amph – amphibole, Gt – goethite, Kln – Kaolinite, Chl – chlorite, Sw. clay – swelling clay, n.d. – not detected, * – traces, + – minor, ++ – intermediate, +++ – major, ++++ – dominant)

Sample number	Qtz	Pl	Kfs	Mca	Cal	Dol	Arg	Amph	Gt	Clay minerals		
										Kln	Chl	Sw. clay
9095	+	+	*	+	++	+	+	+	n.d.			
9096	+	+	n.d.	+	+++	+	+	+	n.d.	+	+	+
9097	+	+	n.d.	+	++++	+	n.d.	+	n.d.			
9098	+	+	*	+	++	+	n.d.	+	+	+		
9099	+	+	+	++	+	+	n.d.	+	n.d.	+		
9100	+	+	+	++	*	n.d.	n.d.	+	n.d.	+	+	+
9101	+	+	+	++	+	+	n.d.	+	n.d.	+		
9102	+	+	+	++	+	+	n.d.	+	n.d.	+	+	*
9103	+	+	+	++	+	+	n.d.	+	n.d.	+	+	*
9104	+	+	+	+	+	+	n.d.	+	n.d.			
9105	+	+	+	+	*	n.d.	n.d.	+	n.d.	+	+	+
9106	++	+	+	+	*	n.d.	n.d.	+	n.d.	+	+	+
9107	+	+	+	+	+	n.d.	n.d.	+	n.d.	+	+	+
9108	+	+	+	++	+	+	n.d.	+	n.d.	+	+	+
9109	++	++	+	++	+	+	n.d.	+	n.d.	+		
9110	+	+	+	++	n.d.	n.d.	n.d.	+	n.d.	+		

1, 2, 6, 7, and 11) and in an old clay pit near Beli Manastir (location 9) while iron nodules are present in four samples (9096, 9101, 9102 and 9107). The birefringence of samples is low to medium-high while the matrix colour is mainly brown with occasional orange and light brown shading. Fired clay briquettes are mainly orange to light brown in colour with dark brown and grey core in several samples, caused by a short firing process.

5.2.4. Mineralogy

The semi-quantitative mineralogical composition of the sediments determined by XRD analysis is presented in **Table 6**. The common feature of all analysed samples is the presence of quartz, plagioclase, micaceous material and amphiboles whereas K-feldspar is almost ubiquitous. Calcite is determined in almost all samples (except 9110) but its presence varies considerably and it is mostly followed by a minor presence of dolomite. Therefore, samples 9095-9098 collected at locations 1 and 2 are distinguished from the others by their high carbonate content and minor aragonite presence. The presence of clay minerals is characteristic of all samples. More specifically, the treatments conducted on the <2µm fraction of samples with “moderate” and “high” plasticity revealed generally uniform clay mineral composition. Kaolinite, chlorite and swelling clays are present in all samples whereas some mixed layer clay minerals are probably present in the samples 9095, 9096, 9097, 9106, 9107 and 9108. Lower swelling clay mineral content is present in samples 9102, 9103 and 9104. Phyllosilicates

at 7 and 14Å are characteristic of all samples with “low” and “no” plasticity evaluated on-site but detailed clay mineral composition on the <2µm fraction has not been determined for those samples.

6. Discussion

The most challenging task in this study is the appropriate identification of raw material sources used in the past. To resolve this issue, it was necessary to properly analyse both the archaeological ceramic samples and potential raw clayey material. The discussion is therefore focused on the raw clayey material, archaeological ceramic samples and consequently their comparison and insight into the sources and distribution of pottery raw materials used in the past.

6.1. Characteristics of clayey material

The clayey sediments collected in the Baranja region are mainly located in a flood plain of the Drava River where channel migration and changes in flood levels lead to the deposition of silty and clayey sediments (Pikija et al., 1991). The similar origin and sedimentary environment of these clays are reflected in their generally uniform mineralogical composition. However, the samples collected near the Gmajna watercourse differ from the others and may be identified as “calcareous clays” due to their high calcite content which is consistent with previous investigations of clayey sediments in the area (Bognar, 1990). Four samples (9100, 9105,

9106, and 9110) are determined as “non-calcareous clays” due to the absence of carbonate phases. Shell fragments in samples 9095 and 9096 are determined by optical microscopy and additionally confirmed by aragonite presence with XRD analysis. Samples can be classified into different categories based on their physical properties.

Measuring the plasticity of clay is not an easy task. The assessment of plasticity depends on the subjective perception of the potter. Therefore, it is often defined descriptively as the optimal condition of the raw material for vessel making or the “workability” of the material based on the potter’s experience and feeling, without a defined quantitative value (Rice, 1987). Accordingly, observation of the plasticity ranges on-site provided an insight into the physical characteristics of the potential raw material sources. Among all the studied samples, those with the highest plasticity were generally found within a radius of 1 km from the site whereas one was collected in abandoned Petlovac clay pit. The high plasticity of these samples generally corresponds to high shrinkage values probably caused by the higher swelling clay mineral content. These clays sometimes need to be mixed with non-plastic inclusions because the high shrinkage affects the vessel causing the breakage during drying and firing (Rye, 1981; Velde and Druc, 1999). Although the observed plasticity varies significantly among samples, grain size analysis revealed two sediment types (Shepard, 1954). The dominant sediments are silt and silty sands—with no samples classified as “clay”. In particular, the samples with the highest observed plasticity, according to grain size analysis, are generally determined as silt or clayey silt which strongly suggests the underestimation of the clay fraction also observed by Fedotov et al. (2007). More specifically, the non-spherical shape of the clay particles, as well as optical properties and measurement limits, led to a different evaluation and increase of silt fraction (Fedotov et al., 2007; Polakowski et al., 2014; Mako et al., 2017). The comparison of these results, together with the geology in the study area, proves that the clayey raw material is easily accessible and available in significant amounts in the vicinity of the settlement, especially near the Gmajna watercourse, making the ancient landscape suitable for the unhindered production of pottery.

6.2. Technological features

This research shows that ancient potters preferred sandy clay throughout all periods of the past. The quartz-rich raw material was used mainly in the Bronze and Late Iron age, while the use of micaceous raw material was less common, and it was used by the potters from the Neolithic, Mediaeval, and especially the Roman period. A higher proportion of non-plastic inclusions of fine-grained crystalloclasts tends to improve the workability of the clay, resulting in the reduced plasticity and shrinkage and a shorter drying process (Orton and

Hughes, 2013; Rice, 1987). Therefore, it was not necessary to add temper material to this clay. Also, the field research has shown that this raw material is present at shallow depths below the soil surface and that ancient potters probably did not need to refine the clay beyond removing unwanted materials such as roots. In this sense, but also technologically, the potters had a high-quality raw material at their disposal.

However, despite the availability of optimal quality clays, ancient potters made extensive use of tempering material or non-plastic inclusions added to the clay. Temper is usually added to a very fine, plastic clay that is highly sticky (Quinn, 2013) to improve the workability of the clay paste but also to reduce shrinkage, resulting in a better-quality product (Rice, 1987; Velde and Druc, 1999). Among the potters from Jagodnjak, the selection of tempering material was not only related to the knowledge about their influence on the production process, but the choice of temper was also subject to tradition (Stark, 1998; Gosselain et al., 2005). The results of this study show that temper selection was distinctive exclusively by a particular social group, i.e. by different periods of the past. For example, the Neolithic potter added plant material to the clay in order to produce different types of vessels. The curvature of the voids present in the matrix shows that the potter tempered the clay with agricultural by-products called chaff. According to the results of numerous archaeometric analyses of Early Neolithic ceramics throughout the Pannonian Plain and the Balkans (Kreiter et al., 2013b; Spataro and Meadows, 2013; Spataro et al., 2021), it was established that the potters from Jagodnjak used a clay recipe by following a practice characteristic of a very wide geographical area. The reasons for using this type of temper material can be related to techno-functional performance. It is known that the vegetal temper increases plasticity and it may also increase the dry strength of very sandy clays (Rice, 1987). Also, research has shown that vegetal tempering provides lighter vessels, ensures resistance to breakage but meanwhile causes reduced heating effectiveness and less abrasion resistance (Skibo et al., 1989). Despite certain positive effects, the use of chaff was not strictly functional, and according to researchers of early Neolithic pottery, the interpretations are more socially oriented (Kreiter et al., 2013b; Dzhanfezova, 2021; Papadakou et al., 2021). In that sense, ceramic production shows a direct relationship with agricultural activities, throughout agricultural by-products incorporated into ceramics. While the technological tradition of plant tempering is widespread and extremely stable for early Neolithic communities, during the Bronze Age in the Pannonian Plain, the same can be said for grog-tempered ceramics. For example, grog (ceramoclasts) is present in the Bronze age pottery from the Jagodnjak site in various quantities (between 7 and 15%) with sizes between 0.25 and 3 mm. As mineralogical composition and grain size distribution of grog do not vary significantly from

the samples' matrix, it can be presumed that grog was made from the same materials as pottery. According to numerous studies on pottery production and use, it has been established that the use of grog is very common among the Bronze age communities across the Pannonian Plain (Kreiter, 2007; Michelaki, 2008; Sofaer et al., 2012; Žibrat Gašparič and Dolenc, 2015; Kudelić et al., 2018). Some authors emphasize the functional basis of grog tempering which improves the physical and thermal properties of the vessel (Rice, 1987; Velde and Druc, 1999). On the other hand, a smaller amount of grog tempering (less than 15%), as recorded in studied samples, can be an indication of pottery recycling as a part of cultural tradition (De Boer, 1974; Smith, 1989; Chapman, 2000; Brück, 2006; Kreiter, 2007). Moreover, it can be noted that Bronze age potters sometimes combined grog tempering along with smaller amounts of lithoclasts, used for making cooking pots and pots for processing or storing food or drink. Small-sized and thin walled vessels (cups and jugs) were made of non-tempered mostly fine-grained sandy clay. Late Iron age pots selected for this study mainly represent a widespread, distinctive type of pottery, found in most parts of the Central European Celtic world. This group of vessels was graphite tempered, presumably cooking pots (Kreiter et al., 2009; Kreiter et al., 2013a; Žibrat Gašparič and Dolenc, 2015) of dark grey colour with a silver shine. These can be easily recognized by the extremely dark matrix colour caused by the presence of graphite grains but also the medium and coarse rock tempering material. The extensive use of graphite during the Late Iron Age is interpreted exclusively as part of the Celtic cultural tradition. However, published research on the use of graphite also implies functional reasons, for example, graphite improves the vessel's resistance to thermal and mechanical stress, increases thermal conductivity and vessels are more resistant to chemical attack (Kreiter et al., 2013a:173 and references therein). On the other hand, the cultural, as well as the economic role of such raw material should be assumed, since graphite sources in the area of the Pannonian Plain are very rare (Kreiter et al., 2013a), indicating a long-distance trade. Lithoclast-tempering tradition is present in all studied periods except the Neolithic, but it is especially common among pottery from the Mediaeval times, and to a lesser extent in Roman ceramics. Bimodal fabrics and angularity of rock fragments with mainly moderately or poorly sorted grains indicate the intentional addition of crushed rocks to clay. Various amounts of rock fragments are usually added to the clay to improve the physical and especially thermal properties of the vessel (Rice, 1987; Velde and Druc, 1999). In our case, such a recipe was used to make pots that are assumed to have been used for the thermal processing of food. The choice of such tempering, during the Roman times, was also observed in the Medieval times, which indicates a tradition that seems to be predominantly based on functionality, unlike in prehistoric periods.

The firing method represents one of the crucial moments in the production process, and it is the final test for a potter. Diagnostic features of firing techniques are the surface colour and the colours of the radial section of the walls (Rye, 1981; Rice, 1987; Roux, 2019). Prehistoric pottery was fired together with the fuel mainly on open fires or in shallow pits. Studied pottery from the Neolithic was fired in the atmosphere of incomplete or complete oxidation indicating uncontrolled firing conditions probably on an open fire. During the Bronze Age, it can be seen that the firing regime depends on the vessel's shape (see Table 1) and it is presumed that pottery was fired on an open fire although kiln firing for fine vessels is not excluded. From the Late Iron Age, onward, kiln firing became the main firing method. Birefringence of the matrix is relatively high for all the studied ceramics, indicating firing temperatures lower than 800-850°C (Quinn, 2013) which is consistent with the firing temperatures usually evaluated for prehistoric pottery. Despite the low birefringence, similar firing temperatures can be assumed for graphite-tempered pottery since the firing temperatures of these wares had to be kept below 850°C in order to avoid accidental oxidation and the loss of graphite (Gebhard et al., 2004). The assessment of firing temperature can also be related to the mineral phases present in a specific temperature range. The presence of calcite and dolomite together with 10Å phyllosilicates, which corresponds to a dehydroxylated illite-like phase, additionally confirms firing temperatures lower than 800°C (Gruver, 1950; Guggenheim et al., 1987; Cultrone et al., 2001; Rodriguez-Navarro et al., 2009; Šegvić et al., 2012; Cultrone and Carrillo-Rosúa, 2020). The lowest firing temperatures can be estimated at around 550-600°C, assumed by recorded cracked quartz grains attributed to the quartz inversion at 573°C (Žibrat Gašparič and Dolenc, 2015 and reference therein). Since the occurrence of cristobalite, as high temperature SiO₂ polymorph, suggest firing temperatures generally above 1200°C, certain peaks present in some samples are more likely the result of SiO₂ glass decomposition, which occurs at lower temperatures (Pagliari et al., 2013).

Technological features, especially those related to the selection of tempering material in prehistory, above all, represent a social signature characteristic of an individual community. On the other hand, the choice of exclusively mineral tempering is characteristic of later, Roman, and Mediaeval communities with interpretations emphasizing function, not the social implications of potters' choices.

6.3. Availability of pottery raw materials

XRD analysis is commonly used and very useful in determining the mineralogical composition of the samples. However, in this research, the XRD analysis did not point to different types of clayey material present in ceramic samples. The possible reason is that tempering

material when mixed with the clay matrix, strongly affect the entire mineralogical composition, and neglect the differences among the clayey materials. Applying the petrological studies is therefore necessary to emphasize the difference between the compositions of clay matrix and tempering material to identify the raw material preferences related to a specific technology, i.e. social group (Rice, 1987; Wallis et al., 2015). The study results point to several potential raw material sources exploited by ancient potters. While collected clays are generally uniform in their mineralogical composition, two general types of raw materials (quartz-rich and micaceous) were determined in the archaeological ceramic samples. In particular, all of the clayey samples collected on site can be approximately correlated with quartz-rich fabric groups (see Figure 6) while three micaceous fabrics remain separated. Despite their similar mineralogical composition, samples can be more precisely separated and correlated with each other by observing the matrix microtexture with the help of optical microscopy. Thus, by comparing the collected clay with the archaeological ceramic matrix, four sources of pottery raw material were located. Even though good quality clayey material is easily accessible near the archaeological site (locations 1, 2, and 4), it is not classified as the raw material used during the studied periods. In contrast, it is assumed that clayey material found at more distant locations, around 4 km from the site was more commonly used (locations 6, 7, 8, and 10). The correlation between pottery made by different social groups and the collected potential raw material indicates several important issues. It seems that most raw material sources do not correspond with a specific pottery tradition but were exploited by different communities from the past. These indicate the intentional choice of quartz-rich raw material throughout all the studied periods. Moreover, the Bronze age and the Neolithic communities used raw material with very common to abundant (35–50%) crystalloclast inclusions in the matrix, which correlate with the clayey material from locations 6, 7, and 8. These can suggest the preferred exploitation of specific raw material sources with a higher amount of crystalloclast inclusions in the matrix. It seems that the clay source location in Petlovac (location 10) with a distance of more than 4 km was less commonly used in prehistory and can be related especially to the Roman period community. Moreover, since all of the clayey sediments collected on site belong to the quartz-rich raw material, the field sampling did not reveal a source of micaceous and inclusion-poor clay (<5% of crystalloclast inclusions) present in the fabrics 8, 9, 10 and 5, respectively. According to the previous investigations of the area (Bognar, 1990), the micaceous clay is probably of local origin but located at specific, still unknown locations. Conversely, the inclusion-poor clay determined as Fabric 5 can be an indication of a long distant transport present among Roman period communities. If excluding this sample, according to the

mineral composition and matrix microtexture, the local origin of pottery can be assumed for all studied samples. Moreover, the frequent occurrence of lithoclasts, predominantly composed of metamorphic rocks which are present at the pre-Neogene Drava basement (Pikija et al., 1991), additionally indicate the local origin of pottery. In the case of graphite tempered pottery from the Late Iron age, a long-distance trade is presumed. Since the sources of graphite in the area of the Pannonian Plain are very rare, it is assumed that such raw material was acquired through trade or exchange (Kreiter et al., 2013a). However, in the case of potters from Jagodnjak, the use of regional graphite is assumed, since the nearest source is located in the Papuk and Psunj mountains (Šinkovec and Krkalo, 1994; Drnić and Mileusnić, 2012), located around 90 km from the settlement where pottery production probably took place.

So where did the potters collect the raw material and how did they use their landscape in terms of pottery production? The use of local clay and easily available tempering materials through all the studied periods is undeniable. However, it is a bit surprising that the potters did not use the clay available at the watercourse, immediately western of the settlement. A discovery of this kind may reflect environmental dynamics, where erosion and alluviation can easily alter the landscape. Therefore, although currently available, these locations may not have been accessible in the past. In addition, the reasons may be hidden in the fact that the area along today's Gmajna Stream represented a kind of boundary or restricted use zone. For instance, according to many ethnographic studies, the resource area in which potters obtain their resources to make pottery may depend on the distribution of their political relationships or it can depend on the social and community boundaries, rather than the geographical distribution of the resources (Livingstone Smith et al., 2005).

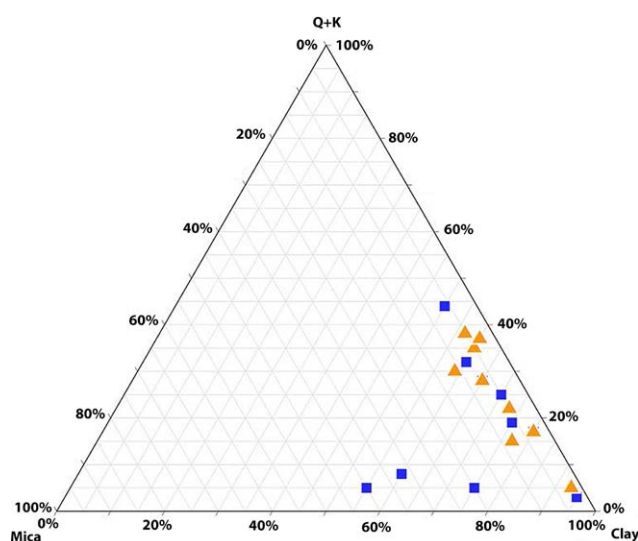


Figure 6: Mineral composition of archaeological ceramic (squares) and clayey material (triangles) fabric groups

7. Conclusion

This paper presents an analysis of the petrography and mineralogy of archaeological ceramics and potential raw materials collected in the vicinity of the multi-period archaeological site (Neolithic, Bronze Age, Late Iron Age, Roman and Medieval period) Jagodnjak-Krčevine located in the lowland region of Baranja. The research objective was to examine the availability and quality of clays for pottery production and to study their distribution in the local landscape. The compositional analysis of archaeological ceramics and the comparison with the clay samples, within the framework of the applied analytical methods (optical microscopy, XRD, grain size analysis), suggests that the pottery raw material (clay and added tempering material) used in the past was of local origin. High quality and diverse clay material is easily available and widely distributed in the local landscape, and interestingly, ancient potters preferred sandy clay in all periods of the past. In this sense, the research showed that the nearest source of quality raw material is only a hundred meters away, but it was not used as a pottery raw material in the past. Also, the study showed that the most likely clay sources used were located to the south and east of the settlement, at a maximum distance of 4 km from the settlement. Moreover, quartz-rich raw material was mainly used during the Bronze and Late Iron Age, while the use of micaceous raw material was less common, and it can be related to the Neolithic, Mediaeval, and especially the Roman period. According to these results, it seems that most ceramics can be associated with sources within a 4 km radius, which is a preferred source especially for Bronze Age potters, but also for Neolithic potters. This indicates patterns in the choice of spatial zones for the procurement of raw materials characteristic of a certain period of the past. On the other hand, the selection of a particular type of tempering material, which has a positive technological effect on shaping, drying, and firing, was preferred by a particular social group and represents a characteristic feature of that community. Thus, Neolithic potters added plant material (chaff) to clay, following a practice that was widespread over a very large geographic area, while tempering with grog is characteristic of the Bronze Age. Celtic potters (Late Iron Age) preferred graphite tempering, suggesting the trade of raw material over long distances, while rock tempering was common in the Roman and Medieval periods for the production of cooking pots. These results represent the first research in Croatia focused on a discussion about distances that potters travelled to obtain their resources in different periods of the past, which, in general, can contribute to the study of a regional system of production and landscape use.

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SAŽETAK

Lončarska tehnologija kroz vrijeme: Arheometrija keramike i lončarske sirovine s višeperiodnog arheološkog nalazišta istočne Hrvatske

Razmatranje višeperiodnih arheoloških nalazišta s ciljem razumijevanja mehanizama kulturnih promjena velikih razmjera još je uvijek vrlo rijetka tema istraživanja u Hrvatskoj. Tehnološke tradicije od velike su važnosti, posebice u kontekstu razmatranja kontinuiteta, inovacija i promjena. U ovom radu upotrijebili smo arheometrijski pristup za istraživanje lončarske tehnologije. Stoga ovaj članak predstavlja analizu petrografije i mineralogije arheološke keramike i potencijalnih sirovina prikupljenih u blizini višeperiodnog arheološkog nalazišta (od neolitika do srednjeg vijeka) Jagodnjak-Krčevine smještenog u istočnoj Slavoniji, tj. jugozapadnom dijelu Panonskog bazena. Primarni cilj je utvrditi kakvu su recepturu lončarske smjese (gline i primjese) koristili lončari za izradu posuda, radi boljeg razumijevanja njihove varijabilnosti u kontekstu tehnofunkcionalnih značajki. Dodatni cilj je ispitati dostupnost i kvalitetu gline za proizvodnju posuda te proučiti njihovu rasprostranjenost u lokalnom krajoliku. Analitičke metode primijenjene u istraživanju su optička mikroskopija, rendgenska difrakcija i granulometrijska analiza. Usporedbom arheološke keramike s glinama utvrđeno je da su drevni lončari vjerojatno koristili lokalno dostupne gline, a odabir primjese koja se glini dodavala predstavlja snažan element tehnotradicije, koji je izraženiji kod prapovijesnih zajednica. Nadalje, ovi rezultati predstavljaju prvo istraživanje u Hrvatskoj usmjereno na raspravu o udaljenostima koje su lončari prelazili kako bi došli do svojih resursa u različitim razdobljima prošlosti, što općenito može pridonijeti proučavanju regionalnog sustava proizvodnje i korištenja krajolika.

Ključne riječi:

arheometrija; Baranja; terensko uzorkovanje; lončarska sirovina; lončarska tehnologija

Author's contribution

Natali Neral (Doctoral Student – Assistant, geologist) participated in field research, conducted laboratory and data analyses, and interpreted and presented the results. **Andreja Kudelić** (Research Associate, archaeologist) designed and organized the research, conducted sampling, and performed the interpretation and presentation of the results. **Ana Maričić** (Associate Professor, geologist) participated in the field research and assisted in the presentation of the results. **Marta Mileusnić** (Full professor, geologist) participated in the interpretation of the results.

Paper 2

Neral, N., Kudelić, A., Maričić, A., Mileusnić, M. (2024) Tracing the origin of raw materials used for the production of ancient ceramics: Case study of multi-period archaeological sites in the Turopolje area (Continental Croatia). Geologia Croatica, 77(3), 269–288. doi: 10.4154/gc.2024.1

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 Čiće-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, provenience

1. INTRODUCTION

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 socio-cultural traditions in the past. For these reasons, the regionally significant archaeological site Staro Čiće-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 (KUDELIC 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

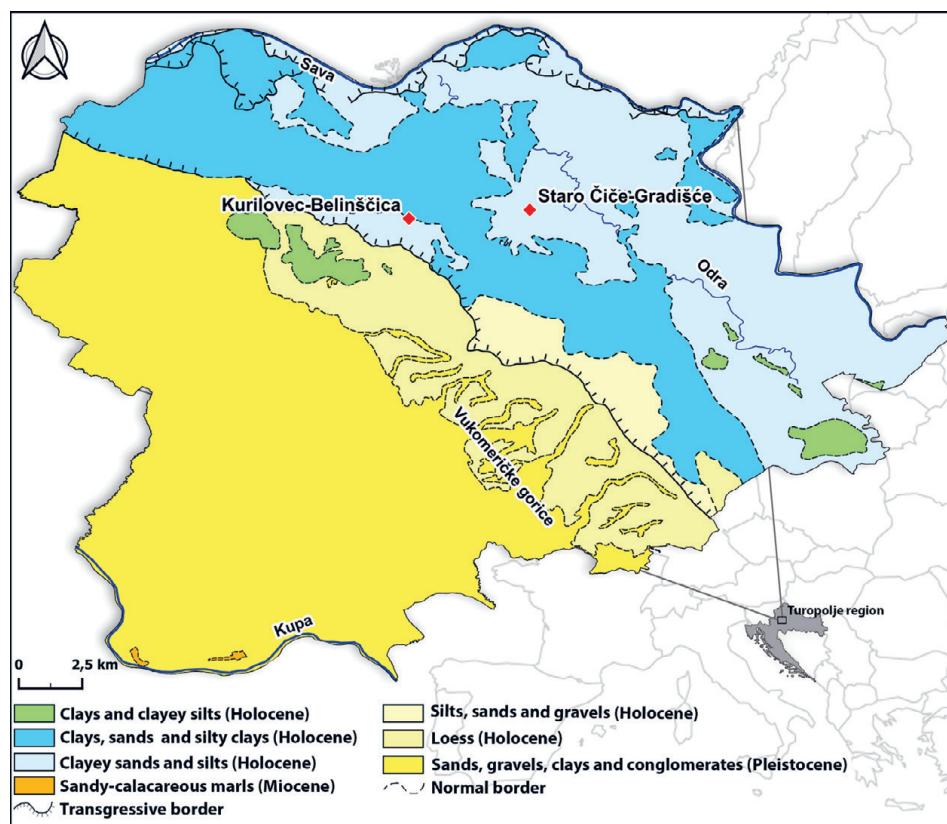


Figure 1. The location and geological map of the Turopolje region with locations of investigated archaeological sites (modified after: PIKIJA, 1987b; BASCH, 1981; KUDELIC & SIROVICA, 2022).

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 multi-period 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 fine-grained unconsolidated deposits like gravels, sands, sandy-clays 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.

Table 1. List of pottery samples from the Staro Čiče-Gradišće and Kurilovec-Belinščica sites selected for analytical methods (/ – unknown vessel type, OM – optical microscopy, XRPD – X-ray powder diffraction, ICP-ES – inductively coupled plasma-emission spectrometry, ICP-MS – inductively coupled plasma-mass spectrometry).

Sample number	Vessel type	Period	OM	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	+	-	-
269	/	Copper/Early Bronze Age	+	+	-
270	/	Copper/Early Bronze Age	+	-	-
271	bowl	Copper/Early Bronze Age	+	-	+
272	/	Copper/Early Bronze Age	+	-	-
273	bowl	Copper Age	+	-	-
274	bowl	Copper Age	+	+	-
275	/	Copper Age	+	-	-
276	/	Copper Age	+	-	-
277	/	Copper Age	+	-	+
278	/	Copper Age	+	-	+
279	/	Copper Age	+	+	-
280	/	Copper Age	+	-	+
281	/	Copper Age	+	-	-
282	/	Copper Age	+	+	-
283	/	Neolithic	+	+	-
284	pot	Neolithic	+	-	+
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	-	-	+
14135	/	Middle/Late Bronze Age	-	-	+
14138	/	Middle/Late Bronze Age	-	-	+
14139	tripod pan	Middle/Late Bronze Age	-	-	+
14144	bowl	Middle/Late Bronze Age	-	-	+
14146	bowl	Middle/Late Bronze Age	-	-	+
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	-	-	+

Staro Čiče-Gradišće

Kurilovec-Belinščica

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-K α radiation. Scan settings were 3–70 °2 θ on the rotating sample stage, 0.013 °2 θ 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 semiquantitative 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 μ 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,

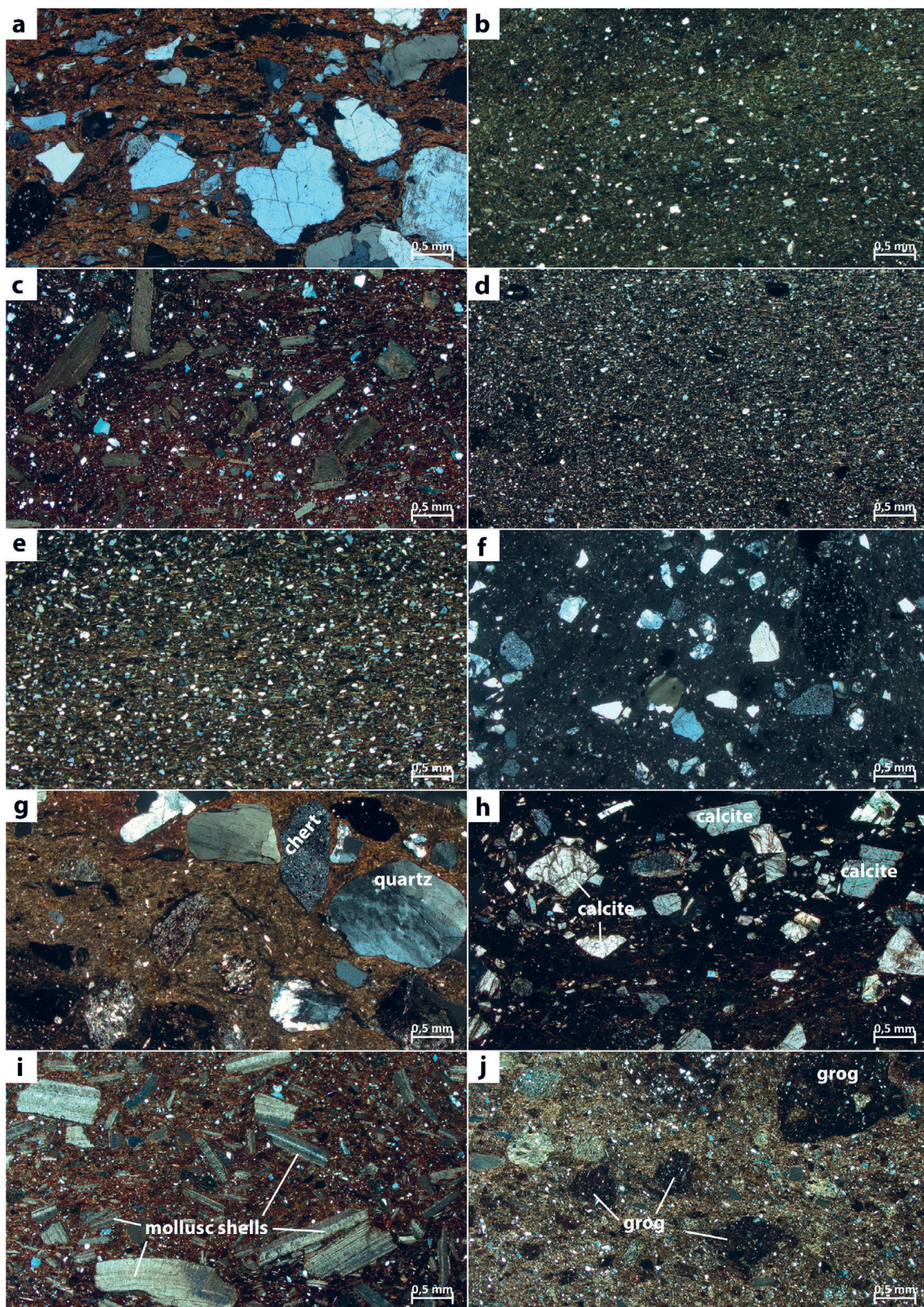


Figure 3. Thin section photomicrographs of ceramics from the Staro Čiče-Gradišće site. (a) Fabric group 1, sample 291, (b) fabric group 2, sample 285, (c) fabric group 3, sample 279, (d) fabric group 4, sample 283, (e) fabric group 5, sample 286, (f) unclassified overfired sample 254, (g) lithoclast 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 sub-angular 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-

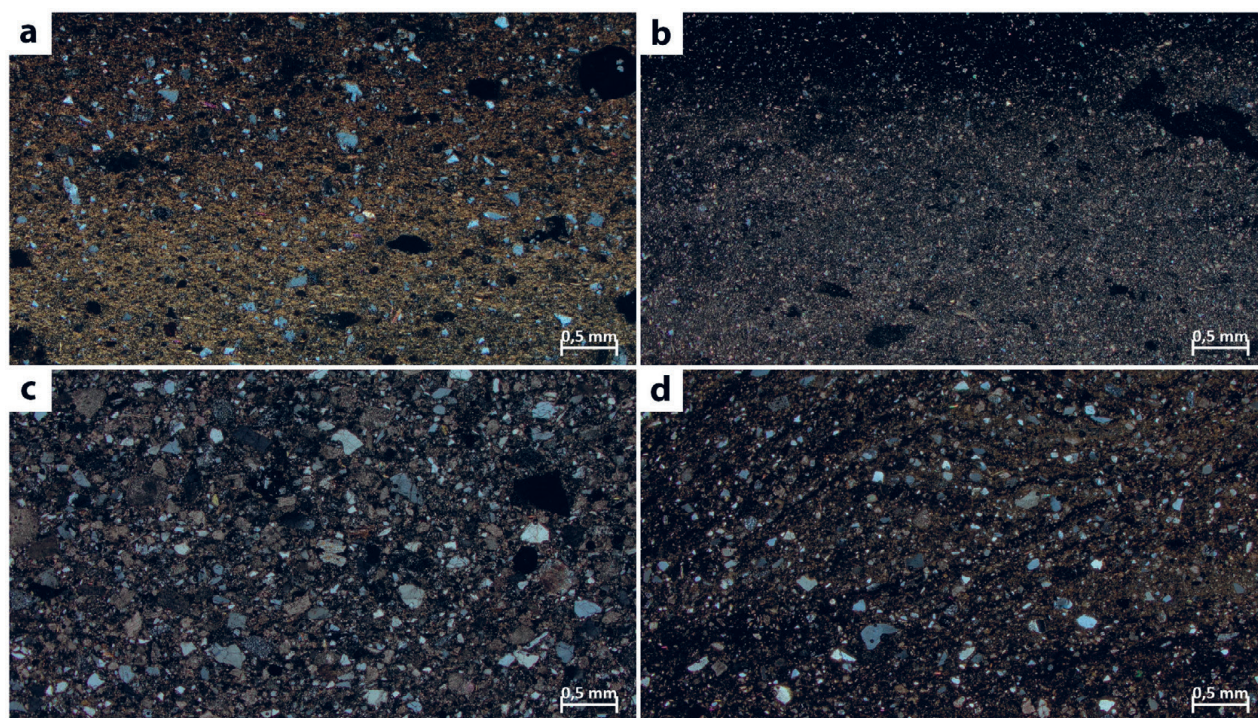


Figure 4. Thin section photomicrographs of fired clay briquettes; (a) sample 102, (b) sample 105, (c) sample 107 and (d) sample 108.

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, A – angular, VA – very angular, SR – sub-rounded, + – present, ++ – dominant, / – unable to estimate the amount of crystalloclast in the matrix since the samples are overfired.

Sample number	Raw clay					Temper material										Fabric subgroups				
	Optical activity	Very fine crystalloclasts (%)	Fine crystalloclasts (%)	Quartz	Feldspar	Muscovite	Biotite	Temper amount (%)	Temper size	Temper sorting	Temper roundness	Metamorphic and igneous fragments	Sedimentary fragments	Calcite	Mollusc shells		Grog amount (%)	Grog size	Grog sorting	Grog roundness
252	H			+	+	+	+	30	F/C	MS	SA/SR	+	++	-	-	-	-	-	-	1.1
253	MH			+	+	+	-	25	F/C	MS	SA/SR	++	+	-	-	-	-	-	-	1.1
255	MH			+	+	+	-	30	VWS	MS	SA/SR	+	++	-	-	-	-	-	-	1.1
259	H			+	+	-	-	40	F/M	VWS	SR	+	++	-	-	-	-	-	-	1.1
266	MH			+	+	+	-	20	M/C	PS	SR	++	+	-	-	-	-	-	-	1.1
268	MH			+	+	+	-	15	M/C	MS	SA/SR	++	+	-	-	-	-	-	-	1.1
278	H	<10		+	+	+	-	-	-	-	-	-	-	-	-	25	MC	WS	SA	1.2
280	H			+	+	+	-	7	M	WS	A	+	-	-	+	15	MC	MS	SA	1.3
281	H			+	+	+	+	30	F/VC	PS	A	+	-	-	+	10	MC	WS	SA	1.3
289	H			+	+	+	-	30	M/C	MS	SA/SR	++	+	-	-	-	-	-	-	1.1
290	H			+	+	+	+	40	F/C	PS	SA	+	-	-	-	-	-	-	-	1.1
291	H			+	+	+	+	30	F/C	PS	SA/SR	+	-	-	-	-	-	-	-	1.1
256	MH			+	+	+	-	25	M/C	PS	SA/SR	++	++	-	-	-	-	-	-	2.1
264	MH			+	+	+	-	20	M/VC	PS	SR	++	-	-	-	-	-	-	-	2.2
273	H			+	+	+	-	30	F/C	WS	A	-	-	-	+	-	-	-	-	2.3
274	MH			+	+	+	-	30	F/C	WS	A	-	-	-	+	-	-	-	-	2.3
275	MH			+	+	+	-	20	F/C	MS	A	-	-	-	+	-	-	-	-	2.3
276	MH	15 – 25	3 – 10	+	+	+	+	10	F/M	WS	A	-	-	-	+	15	M/C	MS	SA	2.4
277	MH			+	+	+	+	20	F/C	PS	A	-	-	-	+	-	-	-	-	2.3
282	MH			+	+	+	-	15	F/M	WS	A	-	-	-	+	30	F/C	MS	SA	2.4
285	MH			+	+	+	-	-	-	-	-	-	-	-	-	-	-	-	-	2.5
288	MH			+	+	+	-	25	F/VC	PS	VA	-	-	++	-	-	-	-	-	2.6
261	H			+	+	+	+	-	-	-	-	-	-	-	-	30	M/VC	PS	SA	3.1
262	MH			+	+	+	+	-	-	-	-	-	-	-	-	40	M/VC	PS	SA	3.1
263	MH			+	+	+	-	20	M/VC	PS	SA	++	+	-	-	-	-	-	-	3.2
265	MH			+	+	+	+	15	M/C	MS	SA	++	+	-	-	-	-	-	-	3.2
267	H	20 – 30	10 – 15	+	+	+	-	25	F/C	MS	SA/SR	+	+	-	-	-	-	-	-	3.2
269	MH			+	+	+	-	-	-	-	-	-	-	-	-	25	M	WS	SA	3.1
270	MH			+	+	+	-	-	-	-	-	-	-	-	-	20	M	WS	SA	3.1
271	MH			+	+	+	-	-	-	-	-	-	-	-	-	15	M	WS	SA	3.1
272	MH			+	+	+	-	-	-	-	-	-	-	-	-	25	M/C	WS	SA	3.1
279	H			+	+	+	-	30	F/C	PS	A	-	-	-	+	-	-	-	-	3.2
258	MH			+	+	+	+	15	F/M	WS	SA/SR	+	++	-	-	-	-	-	-	4.1
260	L			+	+	+	-	-	-	-	-	-	-	-	-	-	-	-	-	4.2
283	H	40	3 – 10	+	+	+	+	-	-	-	-	-	-	-	-	-	-	-	-	4.2
284	MH			+	+	+	+	-	-	-	-	-	-	-	-	-	-	-	-	4.2
287	MH			+	+	+	-	-	-	-	-	-	-	-	-	-	-	-	-	4.1
286	H	40	15	+	+	+	-	-	-	-	-	-	-	-	-	-	-	-	-	5.1
254	L	/	/	+	+	+	-	25	M/C	WS	SA	++	-	-	-	-	-	-	-	/
257	L			+	+	+	+	25	F/C	MS	SA/SR	++	+	-	-	-	-	-	-	/

Table 3. Petrographic analysis of clayey sediments collected in the vicinity of the Staro Čiče-Gradišće archaeological site (MH – medium-high; + – present).

Sample number	Optical activity	Very fine crystallo-clasts (%)	Fine crystallo-clasts (%)	Quartz	Feldspar	Muscovite	Biotite	Metamorphic and igneous fragments	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 sub-rounded. 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 µm fraction revealed differences in the clay mineral compositions. Samples 102, 103, 104 and 108 (Fig. 5) are

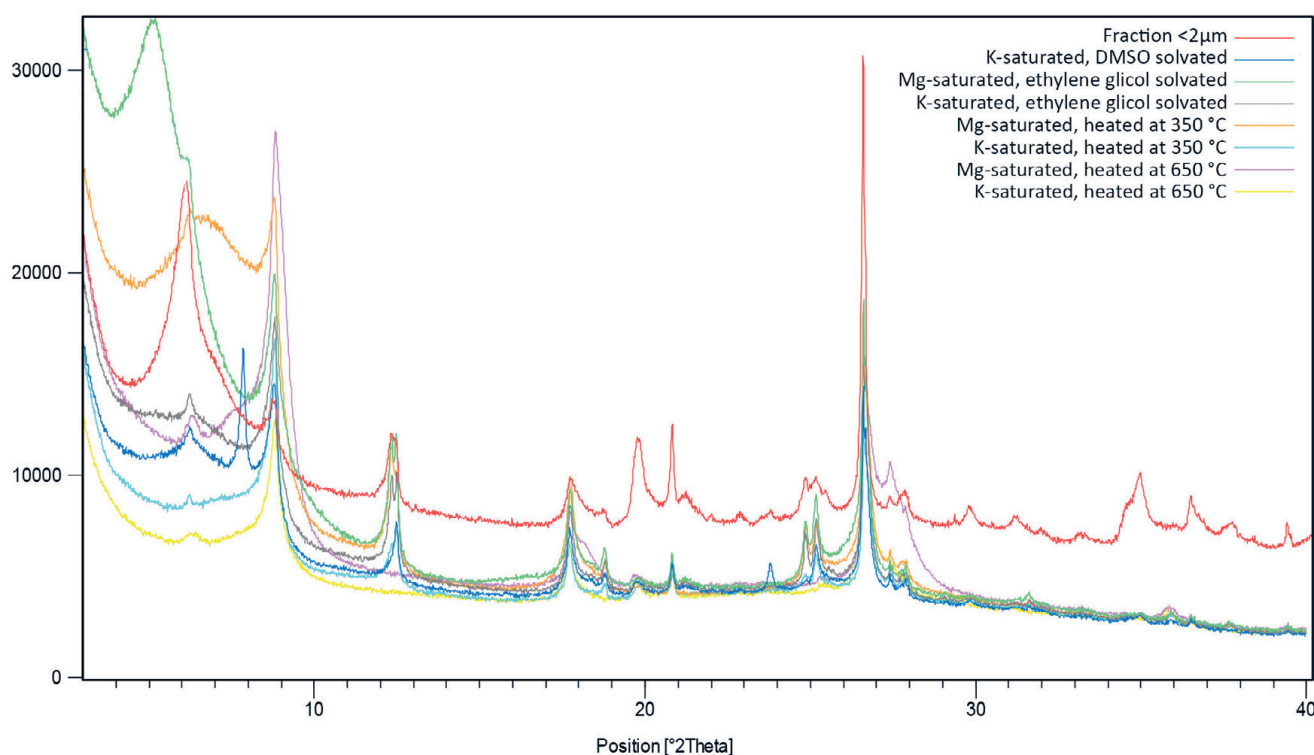
**Figure 5.** X-ray powder diffraction patterns of clayey sample 108.

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	Pl	Kfs	Mca	Cal	Dol	Amph	Hem/Gt	Sp	Ant/Rt	Kaol	Chl	Sm, Vm
Ceramics	255	+	+	+++	+	+	*	-	-	-	-	-	-	-
	256	++	+	+	+	+	*	-	-	-	-	-	-	-
	259	+	+	+	++	+	*	-	-	-	-	-	-	-
	260	+	+	++	+	*	-	-	-	-	-	-	-	-
	262	+	+	+	+	-	*	-	-	-	-	-	-	-
	263	+	++	+	+	+	-	-	-	-	-	-	-	-
	267	++	+	+	+	*	*	-	-	-	-	*	*	-
	269	++	-	+	+	-	-	-	-	*	*	-	-	-
	274	*	+	-	+	+++	*	-	-	-	-	*	*	-
	279	+	-	+	+	+	-	-	*	-	-	-	-	-
	282	+	-	+	+	+	-	-	-	-	-	*	*	-
	283	++	+	+	+	-	-	-	-	-	-	-	-	-
	285	+	+	+	++	+	-	-	-	-	-	-	-	-
	288	+	+	-	+	+++	-	-	-	-	-	-	-	-
	289	++	+	+	+	*	-	-	-	-	-	-	-	-
	291	+	+	++	+	*	-	-	-	-	-	-	-	-
Clayey material	99	++	+	+	++	+	+	-	*	-	-	+	+	*
	100	++	+	+	++	+	+	-	*	-	-	+	+	*
	101	++	+	+	++	+	+	-	-	-	-	*	*	*
	102	++	+	+	++	-	*	-	*	-	-	+	+	++
	103	++	+	*	++	-	*	-	*	-	-	+	+	++
	104	++	+	+	++	*	*	-	*	-	-	+	+	++
	105	++	+	+	++	*	+	*	-	-	-	+	+	*
	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 with sampling depth, plasticity, Munsell colour and shrinkage values (/ - not measured).

Sample number	Sampling depth (cm)	Plasticity	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; MAKU 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 SiO_2 (~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 SiO_2 in the ceramics is likely controlled by different types of igneous and metamorphic rocks added as a temper. Therefore, the lower SiO_2 is in general recorded in the samples without or with a lesser amount of lithoclast temper (samples 265, 277, 278, 280 and 285). The P_2O_5 content is below 5 % except for sample 14147. This probably reflects post-depositional

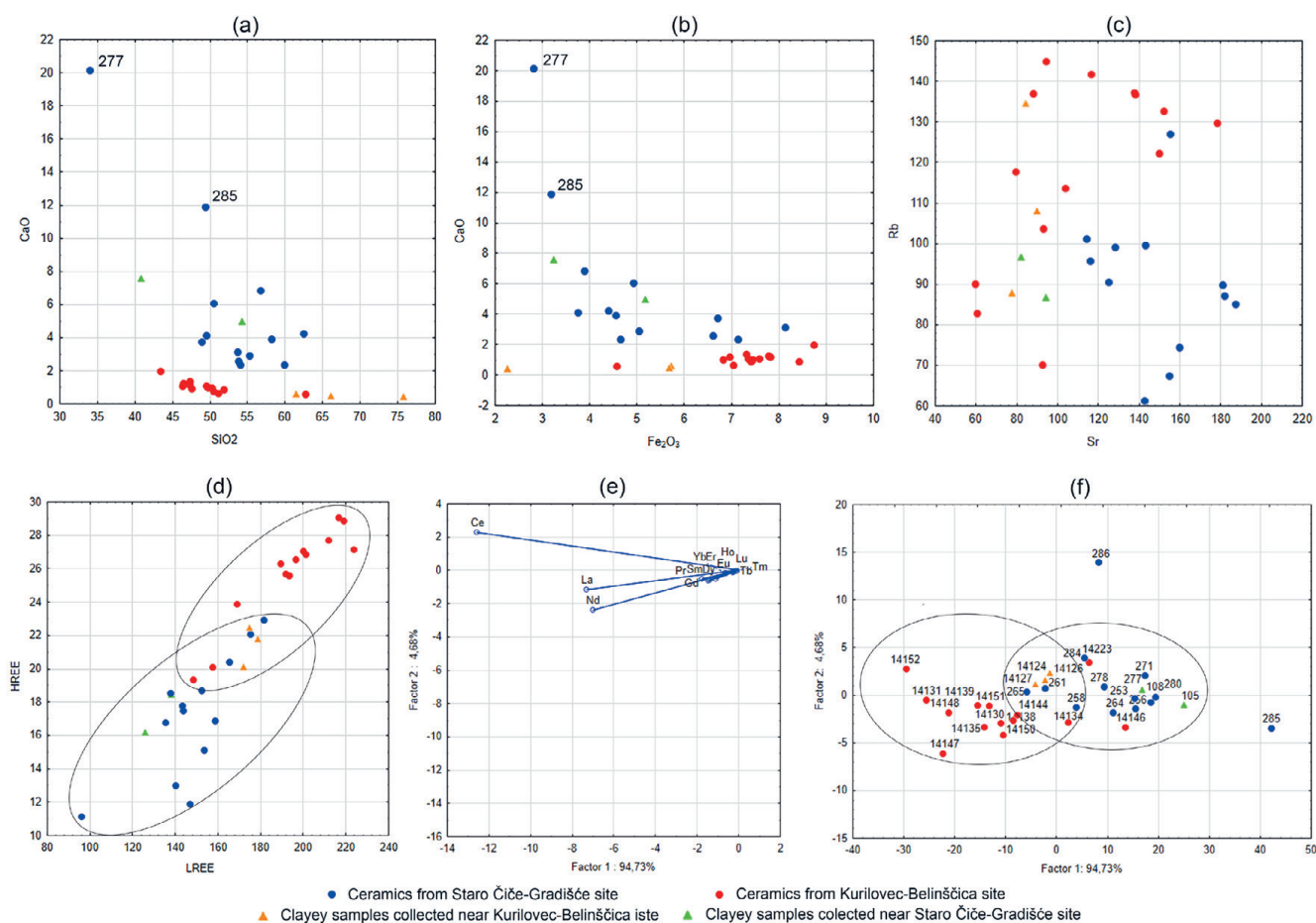


Figure 6. Bivariate plots of (a) SiO_2 vs. CaO, (b) Fe_2O_3 vs. CaO, (c) Sr vs. Rb, (d) LREE (La, Ce, Pr, Nd, Sm, Eu) vs. HREE (Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu), (e) projection of the variables on the factor-plane and (f) principal component analysis based on REE conducted on ceramics from the Staro Čiče-Gradišće 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 Čiče-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 inclusion-rich, 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 thin-sections of pottery from the Staro Čiče-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

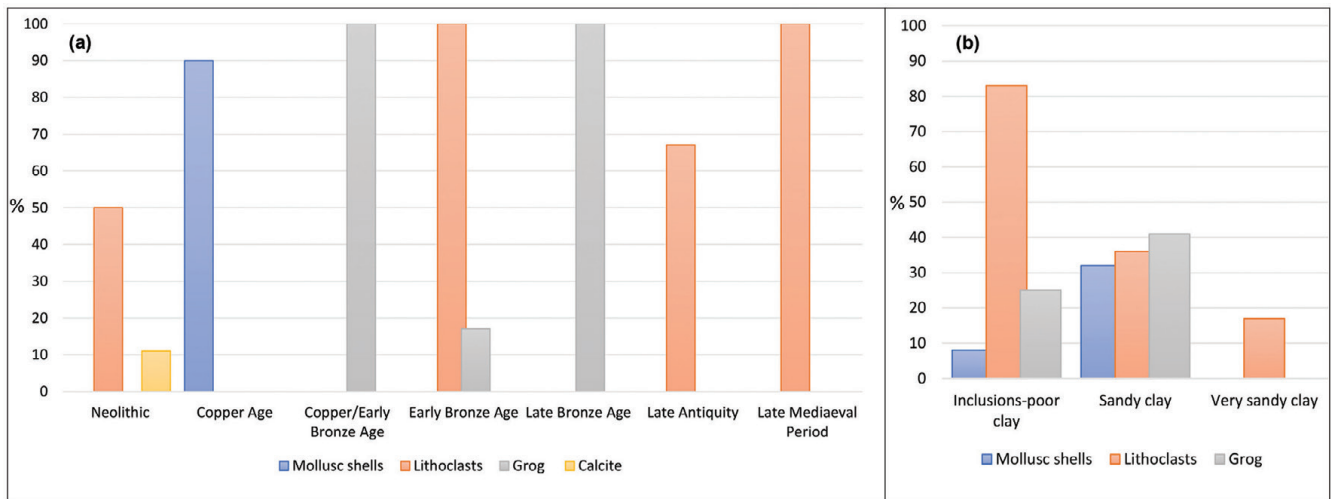


Figure 7. (a) the proportion of different types of tempering material in ceramics from the Staro Čiče-Gradišće site in relation to different periods of the past. (b) the proportion of different types of tempering material in ceramics from the Staro Čiče-Gradišće and Kurilovec-Belinščica site in relation 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 Čiče-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 (KUDELIC et al., 2018; KUDELIC & 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 GAŠPARIĆ, 2004; DOLENEC et al., 2012; SPATARO, 2002; KUDELIC 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 techno-functional 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 Turropolje 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 inclusion-poor 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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Supplement 1. – Geochemical composition of ceramics from Staro Čiče-Gradišće and Kurilovec-Belinščica sites and of clayey samples collected near the sites (SČG – Staro Čiče-Gradišće; K-B – Kurilovec-Belinščica)

Sample number	Sample type	Site	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	MgO (%)	CaO (%)	Na ₂ O (%)	K ₂ O (%)	TiO ₂ (%)	P ₂ O ₅ (%)	MnO (%)	Cr ₂ O ₃ (%)	LOI (%)	SUM (%)
105	clay	SČG	40.78	11.77	3.23	2.88	7.60	0.62	1.68	0.56	0.17	0.02	0.01	30.50	99.87
108	clay	SČG	54.18	13.00	5.16	3.37	5.01	0.81	1.88	0.66	0.12	0.02	0.01	15.60	99.89
14124	clay	K-B	66.10	15.04	5.68	1.12	0.48	0.88	1.92	1.00	0.09	0.07	0.01	7.40	99.84
14126	clay	K-B	75.78	11.86	2.26	0.68	0.44	0.74	1.54	0.98	0.04	0.02	0.01	5.50	99.84
14127	clay	K-B	61.39	16.22	5.71	1.29	0.60	0.76	2.24	0.95	0.15	0.11	0.02	10.40	99.83
253	ceramics	SČG	58.22	17.32	4.56	1.55	3.87	0.59	1.83	0.59	1.78	0.03	0.01	9.40	99.91
256	ceramics	SČG	62.55	15.39	4.41	1.50	4.21	0.65	2.03	0.63	1.30	0.02	0.01	7.10	99.90
258	ceramics	SČG	56.83	14.21	3.89	1.31	6.82	0.76	1.89	0.75	2.63	0.05	0.01	10.60	99.91
261	ceramics	SČG	53.79	17.96	6.61	1.63	2.56	0.69	2.26	0.77	2.51	0.10	0.02	10.80	99.87
264	ceramics	SČG	59.95	18.02	4.66	1.08	2.31	0.42	2.06	0.67	2.52	0.04	0.02	8.00	99.91
265	ceramics	SČG	50.54	17.33	4.93	1.66	6.02	0.72	1.94	0.72	3.12	0.10	0.02	12.60	99.90
271	ceramics	SČG	53.75	16.47	8.15	0.68	3.10	0.38	1.16	1.14	2.61	0.05	0.02	12.30	99.92
277	ceramics	SČG	34.06	13.24	2.83	0.60	20.13	0.24	1.16	0.61	2.93	0.07	0.01	23.90	99.94
278	ceramics	SČG	48.96	19.57	6.72	1.17	3.69	0.14	1.30	0.89	3.03	0.04	0.02	14.20	99.89
280	ceramics	SČG	49.52	22.11	3.76	0.99	4.09	0.20	1.60	0.67	1.70	0.03	0.02	15.10	99.92
284	ceramics	SČG	54.12	18.20	7.15	0.88	2.34	0.56	1.74	1.00	3.05	0.02	0.02	10.70	99.91
285	ceramics	SČG	49.43	11.21	3.20	0.68	11.84	0.59	1.14	0.56	4.17	0.06	0.01	16.90	99.94
286	ceramics	SČG	55.25	17.82	5.05	0.57	2.86	0.14	2.01	0.89	4.61	0.06	0.01	10.50	99.93
14130	ceramics	K-B	47.58	22.23	8.43	1.38	0.87	0.44	2.33	0.85	2.94	0.12	0.02	12.60	99.76
14131	ceramics	K-B	43.41	23.51	8.75	1.30	1.96	0.35	1.42	0.97	2.66	0.10	0.02	15.30	99.79
14134	ceramics	K-B	46.36	22.47	7.60	1.27	1.07	0.46	1.61	0.85	1.90	0.04	0.02	16.10	99.78
14135	ceramics	K-B	46.65	21.73	7.82	1.10	1.16	0.45	2.28	0.90	5.40	0.11	0.02	12.10	99.71
14138	ceramics	K-B	51.88	20.43	7.42	1.40	0.84	0.56	2.13	0.91	2.10	0.06	0.02	12.00	99.77
14139	ceramics	K-B	49.77	22.32	7.45	1.19	0.95	0.45	2.30	0.96	3.80	0.08	0.02	10.50	99.78
14144	ceramics	K-B	46.48	21.14	7.79	1.28	1.25	0.46	2.56	0.86	4.53	0.10	0.02	13.30	99.75
14146	ceramics	K-B	51.13	20.78	7.05	0.78	0.62	0.53	1.56	1.06	0.82	0.03	0.02	15.50	99.83
14147	ceramics	K-B	47.40	19.98	7.32	1.06	1.34	0.49	2.21	0.90	5.26	0.10	0.02	13.60	99.67
14148	ceramics	K-B	49.57	20.29	7.36	1.29	1.03	0.50	2.23	0.90	4.31	0.12	0.02	12.10	99.72
14150	ceramics	K-B	47.37	21.66	6.96	1.53	1.14	0.45	2.15	0.85	3.84	0.05	0.02	13.70	99.71
14151	ceramics	K-B	50.57	21.06	5.15	0.80	0.72	0.29	2.16	0.98	3.68	0.04	0.02	14.30	99.77
14152	ceramics	K-B	50.31	19.78	6.83	0.94	0.96	0.54	2.26	0.96	4.67	0.09	0.02	12.40	99.74
14154	ceramics	K-B	58.69	18.70	4.81	0.67	1.19	0.79	1.57	0.88	0.41	0.07	0.02	12.00	99.84
14160	ceramics	K-B	50.26	22.26	8.12	1.60	0.86	0.34	3.53	0.86	0.54	0.08	0.02	11.30	99.79
14161	ceramics	K-B	46.19	21.96	7.46	0.74	1.56	0.35	1.93	0.84	0.87	0.04	0.02	17.90	99.85
14223	ceramics	K-B	62.75	17.33	4.58	0.59	0.54	0.71	1.69	0.82	0.65	0.10	0.02	10.10	99.86

Supplement 1. – Continued

Sample number	Sample type	Site	Ni (ppm)	Sc (ppm)	Ba (ppm)	Be (ppm)	Co (ppm)	Cs (ppm)	Ga (ppm)	Hf (ppm)	Nb (ppm)	Rb (ppm)	Sn (ppm)	Sr (ppm)	Ta (ppm)
105	clay	SČG	44.00	11.00	426.00	<1	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	0.90
14124	clay	K-B	37.00	14.00	445.00	<1	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	<1	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	<1	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	99.00	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

Supplement 1. – Continued

Sample number	Sample type	Site	Th (ppm)	U (ppm)	V (ppm)	W (ppm)	Zr (ppm)	Y (ppm)	La (ppm)	Ce (ppm)	Pr (ppm)	Nd (ppm)	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

Supplement 1. – Continued

Sample number	Sample type	Site	Tb (ppm)	Dy (ppm)	Ho (ppm)	Er (ppm)	Tm (ppm)	Yb (ppm)	Lu (ppm)	Mo (ppm)	Cu (ppm)	Pb (ppm)	Zn (ppm)	Ni (ppm)	As (ppm)
105	clay	SČG	0.72	4.35	0.87	2.53	0.36	2.38	0.37	0.60	32.00	21.10	72.00	30.30	2.40
108	clay	SČG	0.83	5.08	1.03	2.94	0.41	2.61	0.40	0.70	18.80	18.80	61.00	29.30	9.40
14124	clay	K-B	1.02	5.96	1.18	3.49	0.54	3.38	0.51	0.50	17.10	18.40	60.00	23.90	11.00
14126	clay	K-B	0.90	5.22	1.08	3.39	0.47	3.20	0.52	0.10	9.80	13.30	30.00	11.60	1.80
14127	clay	K-B	1.01	5.81	1.20	3.27	0.49	3.15	0.49	0.50	19.00	27.10	71.00	31.00	10.50
253	ceramics	SČG	0.80	4.71	0.92	2.69	0.39	2.55	0.37	0.30	23.70	16.50	116.00	33.80	3.80
256	ceramics	SČG	0.77	4.59	0.92	2.76	0.34	2.56	0.35	0.30	20.50	16.70	94.00	32.40	3.60
258	ceramics	SČG	0.91	5.35	1.16	3.16	0.45	2.89	0.45	0.40	24.30	15.30	133.00	30.30	4.00
261	ceramics	SČG	0.98	6.04	1.16	3.36	0.47	3.06	0.45	1.10	35.40	21.70	175.00	61.70	12.10
264	ceramics	SČG	0.83	5.07	0.94	2.89	0.40	2.66	0.39	0.30	23.80	17.70	110.00	35.00	6.60
265	ceramics	SČG	1.02	6.21	1.19	3.45	0.46	3.12	0.47	0.40	38.30	19.00	155.00	48.00	12.80
271	ceramics	SČG	0.77	5.15	0.99	3.01	0.44	2.88	0.43	0.60	25.90	19.10	61.00	29.50	11.80
277	ceramics	SČG	0.60	3.54	0.66	1.83	0.24	1.87	0.26	0.20	21.10	16.90	75.00	27.10	7.40
278	ceramics	SČG	0.65	3.93	0.76	2.40	0.35	2.26	0.33	0.50	22.70	23.90	159.00	63.50	10.30
280	ceramics	SČG	0.71	4.44	0.83	2.74	0.38	2.60	0.35	0.10	26.00	18.90	112.00	31.60	10.00
284	ceramics	SČG	0.72	4.36	0.87	2.67	0.36	2.54	0.37	0.20	13.60	20.20	97.00	37.00	40.00
285	ceramics	SČG	0.47	3.07	0.56	1.74	0.23	1.58	0.23	0.40	18.80	12.80	95.00	29.30	9.80
286	ceramics	SČG	0.51	3.18	0.61	1.89	0.26	1.77	0.25	1.00	12.00	25.00	76.00	20.30	20.70
14130	ceramics	K-B	1.22	6.84	1.25	3.77	0.56	3.58	0.53	0.40	56.60	34.80	131.00	79.00	13.60
14131	ceramics	K-B	1.32	7.63	1.48	4.29	0.62	4.16	0.59	0.30	32.60	36.00	88.00	42.10	11.00
14134	ceramics	K-B	1.13	6.38	1.22	3.58	0.50	3.34	0.51	0.40	40.00	28.60	121.00	75.60	11.70
14135	ceramics	K-B	1.25	7.18	1.41	4.07	0.60	3.89	0.56	0.40	26.60	26.40	125.00	44.30	8.30
14138	ceramics	K-B	1.24	6.89	1.32	4.04	0.58	3.63	0.58	0.40	29.20	26.00	110.00	60.30	11.60
14139	ceramics	K-B	1.24	7.03	1.46	3.83	0.61	3.91	0.59	0.30	24.40	27.90	87.00	41.10	9.00
14144	ceramics	K-B	1.18	6.62	1.36	3.80	0.57	3.56	0.58	0.60	38.90	33.00	135.00	62.40	16.00
14146	ceramics	K-B	0.85	5.13	1.03	2.89	0.44	2.82	0.44	0.30	28.10	25.20	60.00	56.60	15.90
14147	ceramics	K-B	1.39	7.56	1.50	4.07	0.62	4.14	0.54	0.40	41.80	28.70	131.00	56.80	10.00
14148	ceramics	K-B	1.34	7.12	1.45	3.98	0.62	4.00	0.61	0.40	36.60	30.30	128.00	59.20	10.70
14150	ceramics	K-B	1.22	6.84	1.33	3.77	0.56	3.55	0.55	0.40	40.00	29.40	147.00	73.60	10.90
14151	ceramics	K-B	1.23	6.96	1.48	4.14	0.59	3.79	0.59	0.20	39.60	27.90	83.00	38.60	11.70
14152	ceramics	K-B	1.23	6.98	1.47	4.17	0.56	3.83	0.58	0.40	31.20	25.80	103.00	50.10	11.90
14154	ceramics	K-B	1.01	5.91	1.22	3.55	0.49	3.20	0.51	0.20	24.10	29.20	68.00	44.00	8.20
14160	ceramics	K-B	1.06	6.08	1.23	3.35	0.51	3.47	0.53	1.20	39.30	39.90	150.00	102.80	9.60
14161	ceramics	K-B	1.17	6.40	1.29	3.69	0.55	3.59	0.53	0.20	36.90	34.80	116.00	59.70	8.20
14223	ceramics	K-B	0.94	5.07	1.08	3.14	0.44	3.06	0.47	0.20	21.20	25.10	67.00	44.20	10.30

Supplement 1. – Continued

Sample number	Sample type	Site	Cd (ppm)	Sb (ppm)	Bi (ppm)	Ag (ppm)	Au (ppm)	Hg (ppm)	Tl (ppm)	Se (ppm)
105	clay	ŠČG	0.50	0.30	0.30	<0.1	2.10	0.13	0.10	5.90
108	clay	ŠČG	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	ŠČG	0.20	0.10	0.30	<0.1	0.80	<0.01	0.30	<0.5
256	ceramics	ŠČG	0.20	0.20	0.10	<0.1	<0.5	0.02	0.30	<0.5
258	ceramics	ŠČG	0.50	0.20	0.10	<0.1	1.00	0.02	0.20	<0.5
261	ceramics	ŠČG	0.80	0.20	0.30	<0.1	1.00	0.04	0.20	<0.5
264	ceramics	ŠČG	0.30	0.20	0.20	<0.1	0.80	0.02	0.30	<0.5
265	ceramics	ŠČG	0.60	0.20	0.20	<0.1	3.00	0.03	0.20	<0.5
271	ceramics	ŠČG	0.30	0.40	0.20	<0.1	1.10	0.02	0.20	<0.5
277	ceramics	ŠČG	0.50	<0.1	0.20	<0.1	1.00	0.03	0.20	<0.5
278	ceramics	ŠČG	0.50	<0.1	0.30	<0.1	1.00	0.03	0.30	<0.5
280	ceramics	ŠČG	0.30	<0.1	0.30	<0.1	1.60	0.02	0.30	<0.5
284	ceramics	ŠČG	0.30	0.10	0.40	<0.1	0.70	0.14	0.20	<0.5
285	ceramics	ŠČG	0.80	0.20	0.20	<0.1	2.20	0.05	0.20	<0.5
286	ceramics	ŠČG	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
14223	ceramics	K-B	0.20	0.20	0.20	<0.1	0.60	0.02	0.20	<0.5

Paper 3

Kudelić, A., Neral, N. (2025) Selection of raw material through the history of pottery production in Istria (Croatia): Social implications of paste variability. Archaeological and anthropological science, 17, 67, 2-24. <https://doi.org/10.1007/s12520-025-02182-x>

Paper 4

Neral, N., Kudelić, A., Maričić, A., Kovačević Zelić, B. (2025) Experimental Study of Physical and Mechanical Properties of Ceramics: Insights into Raw Material Selection in Prehistoric Croatia. Applied clay science, 274, 107859. <https://doi.org/10.1016/j.clay.2025.107859>

3. Discussion

3.1. Properties of the clayey raw material

The analysis of pottery from various geographical regions and historical periods within Croatia has identified a diverse range of raw materials used in ceramic production. Ceramic petrography reveals that potters predominantly worked with clay materials containing 15–40% well-sorted, sub-angular to sub-rounded inclusions, generally occurring in sizes up to 0.25 mm. These inclusions primarily consist of quartz, feldspar, mica, calcite, dolomite while the remaining mineralogical composition is dominated by clay minerals such as kaolinite, chlorite, illite, smectite, vermiculite, and mixed-layered clay minerals. Based on these characteristics, such raw materials can be classified as silty or sandy clay (Fig. 3a). Following the terminology established in previous studies, which define these materials as sandy clay, this study also adopts the term sandy clay to describe this type of sediment (Kreiter, 2007; Kudelić, 2016; Kudelić *et al.*, 2023; Kudelić & Neral, 2025; Neral *et al.*, 2023, 2024, 2025). In contrast, clayey raw materials with a higher proportion of clay minerals and a lower concentration of other naturally occurring inclusions (e.g., quartz, feldspar, mica, calcite, dolomite) are less commonly utilized and are classified as inclusion-poor clays (Fig. 3a). These broad categories can be further subdivided based on specific material properties, ensuring a more precise classification of ceramic raw materials (Fig. 3b).

The use of sandy clays (Fig. 4a), is particularly prominent in pottery from Continental Croatia, encompassing the regions of Central and Eastern Croatia (Jagodnjak-Krčevine, Staro Čiče-Gradišće and Kurilovec-Belinščica sites). This area is characterized by extensive alluvial plains shaped by river systems (Basch, 1983; Pikija, 1987; Šikić *et al.*, 1979), which facilitate the deposition of diverse secondary clay-rich sediments transported from their parent rock sources. In such an environment, sandy clays accumulate under high-energy conditions, resulting in the deposition of coarser particles alongside clay minerals (Velde & Meunier, 2008). Experimental study conducted as part of this doctoral research (Neral *et al.*, 2025) indicate that such clay exhibits moderate plasticity (plasticity index=28%) and shrinkage (~22%), properties that enhance their suitability for ceramic production. Given these advantageous characteristics, it is unsurprising that sandy clays represent the most commonly utilized raw material in prehistoric pottery-making

practices in Croatia (Kudelić *et al.*, 2018; Spataro *et al.*, 2021; Kudelić & Sirovica, 2022; Kudelić *et al.*, 2023; Neral *et al.*, 2023, 2024).

In contrast to the predominantly utilized sandy clays, the use of inclusion-poor clay has also been documented (Fig. 4b), particularly in Central Croatia (Fig. 3b). Inclusion-poor clays typically accumulate under low-energy depositional conditions or as a result of prolonged transport and sorting processes, leading to a higher concentration of fine clay minerals. This type of clay exhibits significantly high plasticity (plasticity index=59%) and shrinkage (~34%), attributes primarily linked to the elevated presence of clay minerals and swelling clay minerals. Swelling clay minerals possess the ability to absorb water into their interlayer spaces, thereby enhancing plasticity. Furthermore, the high concentration of clay minerals increases the overall water adsorption capacity of the material. The negatively charged surfaces of these minerals attract polar water molecules, forming a lubricating layer that facilitates particle movement, thereby improving plasticity. As a result, clays with a greater proportion of fine clay minerals exhibit increased plasticity due to the larger surface area available for water adsorption. This type of clay necessitates prolonged drying periods due to its high clay mineral content, which leads to substantial water loss and increased susceptibility to deformation. In contrast, sandy clays, characterized by a lower proportion of clay minerals, exhibit reduced shrinkage and better stability during the drying process. Despite these challenges, inclusion-poor clays were still utilized in prehistoric pottery production, particularly by Bronze Age communities in lowland Central Croatia (Kudelić *et al.*, 2018; Neral *et al.*, 2024). The results from the experimental study (Neral *et al.* 2025) suggest that ceramics produced from inclusion-poor clays exhibit higher point load strength values (mean 1.41 MPa) than those made from sandy clay (mean 0.79 MPa) due to improved particle packing, aligning with findings from a recent study by Bernasconi *et al.* (2024). These findings indicate that prehistoric communities inhabiting present-day Continental Croatia primarily selected sandy clay due to its accessibility and ease of processing and firing. However, the deliberate use of inclusion-poor clay may indicate a strategic material selection based on its functional advantages. Although inclusion-poor clay required more complex processing and firing techniques, its capacity to produce stronger and more durable ceramics suggests that these communities were probably prioritized material strength and durability over production efficiency.

The types of the clay raw materials utilized in Istria and Central Dalmatia also corresponds with the geological structure of the region (Fig. 3b). These areas are predominantly karstic

environments (Magaš & Marinčić, 1973; Marinčić *et al.*, 1977; Velić *et al.*, 1995; Tišljarić *et al.*, 1998; Vlahović *et al.*, 2005; Velić & Vlahović 2009), characterized by limestones and dolomites, where clayey deposits are relatively scarce and primarily associated with Cretaceous rocks linked to paleo-relief and paleo-karst formations. Analysis of raw materials found in pottery from Istria (Old Rakalj, Šiljar, and Saint Theodore sites) reveals that the majority contain fragments of clay coatings, a common feature of terra rossa polygenetic soils. These fragments form through the pedogenic process of clay illuviation, wherein clay particles are removed from the upper (eluvial) horizons and accumulate in the lower (illuvial) horizons, filling dissolution voids in carbonate rocks in contact with terra rossa (Fedoroff, 1997; Durn, 2003; Poch, 2014; Durn *et al.*, 2023). Furthermore, the geochemical characteristics of these materials, particularly the La/Ce, Sm/Nd, and Ti/Al ratios used to assess the geochemical similarity of elements in silicate parent materials, correspond with those identified in terra rossa soils from the northwestern Istrian Peninsula (Durn *et al.*, 2023; Kudelić & Neral, 2025) and red paleosols from Susak Island (Durn *et al.*, 2018a; b). Therefore, it appears that potters from Istria commonly utilized readily available terra rossa over the centuries, as also reported by Mange & Bezeczky (2006) in their study of Roman amphorae sherds. Characterized by their distinctive red colour due to hematite formation, terra rossa soils contain clay minerals (e.g., Durn *et al.*, 2021; Durn *et al.*, 2023; Kudelić & Neral, 2025), making them well-suited for pottery production. Based on the proportion of clay minerals and other naturally occurring inclusions, the terra rossa found in the majority of ceramic assemblages from Istria can be classified as "sandy terra rossa" (Fig. 3b, 4c). In contrast, a smaller subset of ceramic samples with a significantly higher clay mineral content is designated as "inclusion-poor terra rossa" (Fig. 3b, 4d). The sole exception from the use of terra rossa as the primary raw material was identified in a small group of pottery samples (more details in section 3.2.) containing recrystallized planktonic foraminiferal tests, which are consequently classified as marl (Ćosović *et al.*, 2004, Fig. 3b, 4e).

To ensure broader regional coverage, this study is further enriched by the findings of Kudelić *et al.* (2023; 2024) and the investigation of Bronze Age ceramics from Central Dalmatia and its hinterland. The results demonstrate that Bronze Age potters predominantly employed sandy clay as the primary raw material. Additionally, the occasional occurrence of poorly sorted clayey materials containing fine to medium sized (0.25–1 mm) sub-angular quartz, feldspar, and mica suggests the use of primary clays—residual deposits that have formed in situ and remained at their

location of origin. These are accordingly classified as coarse sandy clay (see Fig. 3b, 4f). A similar pattern of clay raw material utilization can be inferred for other prehistoric periods; however, the limited availability and research of ceramic findings from these time frames results in a restricted dataset. Notably, research on fine tableware production during the Hellenistic period in Central Dalmatia indicates that potters primarily utilized locally available flysch deposits as raw materials. Given the limited occurrence of flysch deposit outcrops, these materials were likely sourced from the same geological formation on Hvar Island and subsequently distributed to multiple manufacturing workshops (Miše *et al.*, 2020).

The analysis of pottery from various historical periods and geographical regions within Croatia has revealed significant differences in the properties of clay raw materials between Continental Croatia (Eastern and Central Croatia) and the Eastern Adriatic coast (Istria and Central Dalmatia). These findings underscore the critical role of environmental factors in shaping technological choices, as the composition and availability of raw materials directly influenced the decisions made by ancient potters. Across all historical periods, potters primarily selected readily available clayey deposits that possessed the necessary properties for pottery production. However, the intentional selection of available highly plastic, inclusion-poor clay by Bronze Age communities in Central Croatia, despite the availability of sandy clay, suggests a strategic decision based on the functional advantages of this material, particularly its enhanced strength and durability.

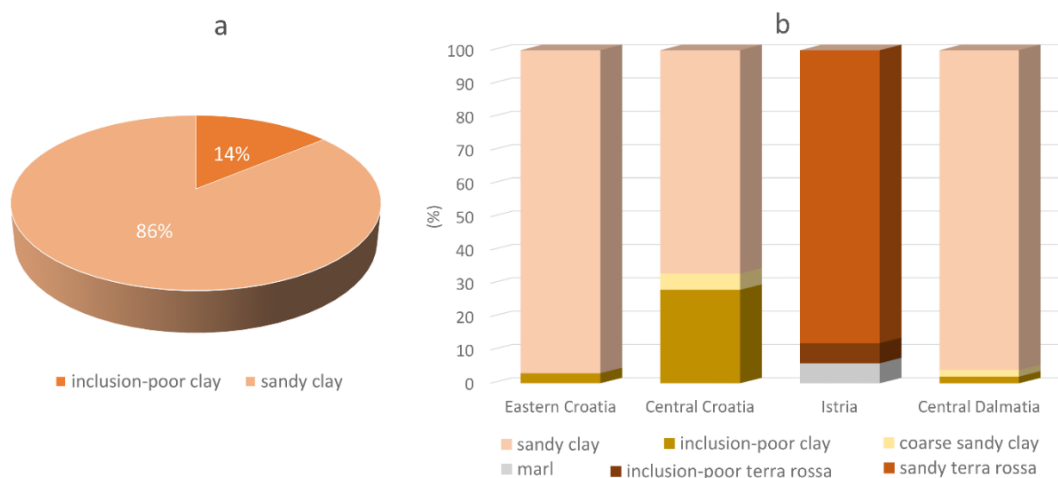


Figure 3. (a) Proportion of sandy and inclusion-poor clay in analysed pottery samples and (b) proportion of specific types of clayey raw materials in individual case studies based on data from Neral *et al.* (2023, 2024), Kudelić & Neral (2025), and Kudelić *et al.* (2023, 2024).

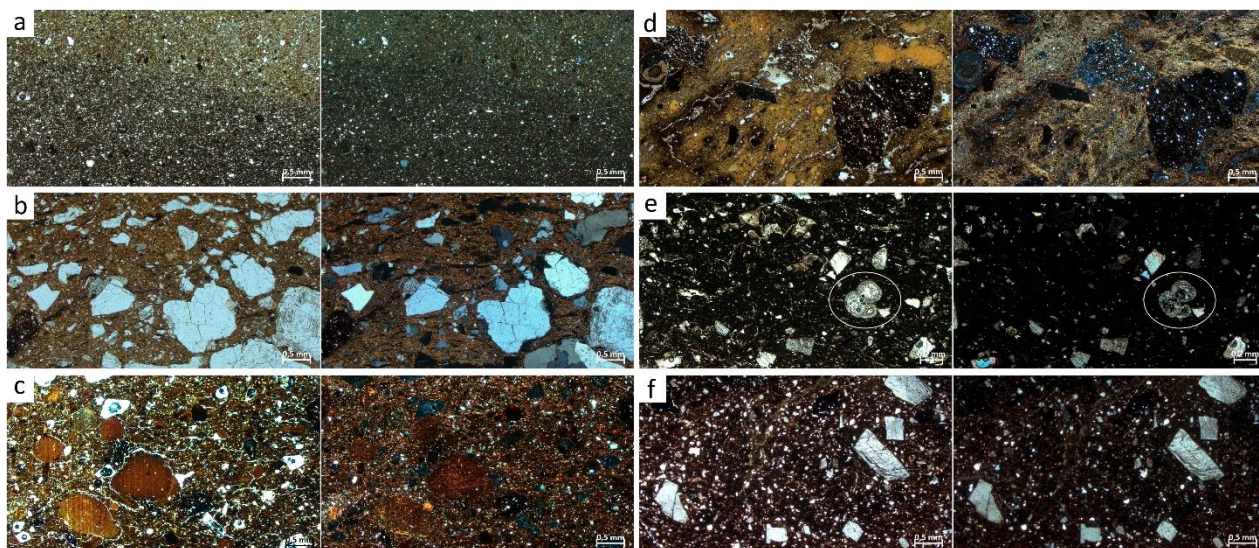


Figure 4. Thin section photomicrographs of identified clayey raw material types in pottery samples. (a) Sandy clay, sample 8989 from the Jagodnjak-Krčevine site (Neral *et al.*, 2023), (b) inclusion-poor clay, sample 291 from the Staro Čiče-Gradišće site (Neral *et al.*, 2024), (c) “sandy terra rossa,” sample 175 from the Šiljar site (Kudelić & Neral, 2025), (d) “inclusion-poor terra rossa,” sample 190 from the Old Rakalj site (Kudelić & Neral, 2025), (e) marl with planktonic foraminiferal tests, sample 203 from the Old Rakalj site (Kudelić & Neral, 2025), and (f) coarse sandy clay from Marina-Drid site, sample 76 (Kudelić *et al.*, 2023). Right microphotograph of each sample is taken in cross polarized light.

3.2. Provenience of the clayey raw material

One of the most significant challenges of this study lies in the precise identification of the raw material sources (i.e., provenience) used in ancient pottery production. A scientific approach to determining the provenience of pottery raw materials requires a comprehensive analysis of both archaeological ceramic samples and potential clay sources from the surrounding environment. The application of appropriate analytical techniques is critical for identifying similarities between raw clay and pottery samples. Geochemical analyses are particularly valuable as they offer detailed insights into the elemental composition of both ceramic and raw clay samples. Complementarily, ceramic petrography provides essential information on textural features and potential correlations between the ceramic material and its clay source. Although geochemical methods yield precise elemental concentrations, identifying the exact location of raw material sources remains challenging. This difficulty arises because similar types of clayey raw materials from different nearby locations often exhibit very similar geochemical compositions. Nevertheless, by employing a comparative method that analyses both ceramics and clays, it is possible to determine the type

of raw material used and estimate the likely radius within which the raw material was sourced. Considering geochemical analysis, the provenience of pottery raw materials has primarily been assessed through the analysis of rare earth element concentrations. This approach is particularly relevant since the concentrations of major and minor elements can be affected by the addition of tempers, whereas REEs are predominantly associated with accessory minerals and are concentrated in clay- and silt-sized fractions (Hein *et al.*, 2004; Carloni *et al.*, 2021).

The identification of similar geochemical patterns between ceramics and clay raw materials collected near the investigated archaeological sites in Central Croatia and Istria suggests that most of the analysed pottery is of local origin, with raw materials likely sourced within a 4 km radius from the place of production (Neral *et al.*, 2024; Kudelić & Neral, 2025). This pattern aligns with the ethnographic observations (Arnold, 1980:149, 1985: 39–49, 2000; Gosselain & Livingstone Smith, 2005), which suggest that potters traditionally acquired resources within a comparable range guided by the principles of minimal time investment and proximity to the production site. However, indicators of "non-local" origins are generally the exception rather than the rule, suggesting the influence of additional factors such as trade, migrations, or other socio-economic dynamics. This research has identified several instances of such non-local origins.

In central Croatia (Staro Čiče-Gradišće site), Neolithic-period samples exhibit distinct REE signatures, setting them apart from the rest of the dataset (Neral *et al.*, 2024). This strongly suggests that potters either sourced raw materials from more distant locations or that exchange networks facilitated the distribution of clay resources among Neolithic communities. A similar pattern is observed in eastern Istria, where the results indicate that potters primarily utilized locally available terra rossa soils as a raw material. However, at the Šiljar and Old Rakalj sites, ceramics containing recrystallized planktonic foraminiferal tests have been identified (Kudelić & Neral, 2025). These microfossils are typically found in marls, with the nearest known deposits located approximately 20 km north of the settlements (Ćosović *et al.*, 2004; Babić *et al.*, 2007; Gulam *et al.*, 2014). Their presence along the right bank of Raša Bay suggests that these materials were likely transported from settlements across the bay or possibly sourced from the interior regions of the Istrian Peninsula. Additionally, certain Bronze age pottery samples with distinct geochemical compositions further support the possibility of trade or exchange networks in the region. Considering the Eastern Croatia, the similarities in physical and microtextural characteristics between ceramics and local clay raw materials suggest that pottery production predominantly

relied on locally available resources. However, due to the absence of geochemical analysis for this dataset, a more detailed discussion on the provenience of raw materials remains inconclusive. Similarly, the examination of mineralogical, petrographical, and textural characteristics of Bronze Age pottery samples from Central Dalmatia and its hinterland (Kudelić *et al.*, 2023, 2024) suggests that raw materials were sourced locally. However, research on ceramic production during the Hellenistic period in Central Dalmatia (4th–1st century BC) reveals both the exploitation of local raw materials and the importation of goods. Specifically, fine tableware was produced using locally sourced raw materials, supplied from the same geological source to various manufacturing workshops (Miše *et al.*, 2020). Conversely, the raw material used for amphora production originated from Greece and Italy, indicating that finished amphorae were imported from these regions rather than being locally manufactured. This observation aligns with the historical context of the period, which was marked by a highly developed maritime trade network (Miše & Quinn, 2022).

3.3. Properties and provenience of tempering material

Consistent with previous studies, the selection of tempers was primarily determined by the physical and mechanical properties they imparted to the finished vessel, optimizing factors such as durability, thermal resistance, and workability. However, in some cases, it was also influenced by specific time periods and deeply ingrained traditions (e.g. Shepard, 1956: 164; Rice, 1987: 118; Stark, 1998; Gosselain, 1999; Gosselain & Livingstone Smith, 1995; Michelaki *et al.*, 2014). This research further reveals that while availability of resources and technological considerations played a primary role in temper selection, social practices, temporal variations, and regional traditions also contributed to the diversity of materials used.

Neolithic

The long duration of the Neolithic period led to variations in tempering practices between its early and later phases. In eastern Croatia, Early Neolithic ceramics from the Jagodnjak-Krčevine site contain agricultural by-products, particularly chaff (Fig. 5a), as evidenced by the characteristic curvature of voids within the ceramic matrix (Neral *et al.*, 2023). This suggests a strong link between ceramic production and agricultural activities, with chaff deliberately added to the clay. Similarly, archaeometric analyses of Early Neolithic ceramics from the Pannonian Plain (Kreiter

et al., 2013; Spataro & Meadows, 2013; Spataro, 2019; Spataro et al., 2021), and to a lesser extent from the Adriatic coast (Spataro, 2002), indicate the use of vegetal material as temper. This suggests that potters followed a widely shared clay preparation tradition across these regions. However, experimental study (Neral *et al.*, 2025) suggest that the addition of vegetal material does not enhance the mechanical strength of ceramics compared to non-tempered samples. While vegetal tempering increases plasticity and may improve the dry strength of highly sandy clays (Rice, 1987), previous research has shown that it results in lighter vessels with greater resistance to breakage but lower thermal efficiency and abrasion resistance (Skibo *et al.*, 1989; Quinn, 2022: 219). Consequently, while chaff tempering may have offered certain functional advantages, its use was likely driven by social or symbolic considerations rather than purely utilitarian factors (Kreiter *et al.*, 2013; Dzhanfezova, 2021; Papadakou *et al.*, 2021). Notably, the practice appears to have been gradually abandoned by the end of the Early Neolithic, indicating a shift in ceramic production techniques and material selection.

Conversely, Neolithic pottery from Central Croatia, specifically from the Staro Čiče-Gradišće site, demonstrates a distinct tempering tradition. Petrographic analysis has identified the presence of lithoclasts, including fragments of sedimentary, metamorphic, and igneous rocks (Neral *et al.*, 2024, Fig. 5b). The diversity of lithoclast types and their sub-rounded shapes suggests that these materials were derived from natural sand deposits, most likely sourced from nearby riverbeds or alluvial deposits. This pattern indicates a technological preference for the utilization of locally available raw materials consistent with broader regional practices documented in Central Croatia (Spataro, 2019; Spataro *et al.*, 2021). A comparative analysis of regional tempering practices highlights notable variations. Middle and Late Neolithic communities along the Adriatic coast predominantly employed mineral tempers, such as limestone and crushed calcite (Spataro, 2002; Žibrat Gašparič, 2004; Spataro & Meadows, 2013; Bernardini *et al.*, 2024), whereas Late Neolithic communities in present-day Hungary primarily utilized grog as a tempering material (Kreiter *et al.*, 2013; 2017). The identification of calcite temper in a single Neolithic sherd from Staro Čiče-Gradišće (Neral *et al.*, 2024) implies either the use of non-local raw material sources or the existence of exchange networks that enabled the distribution of ceramic production materials among Neolithic communities, given that calcite is not commonly found within the geological context of the region (CGS, 2009).

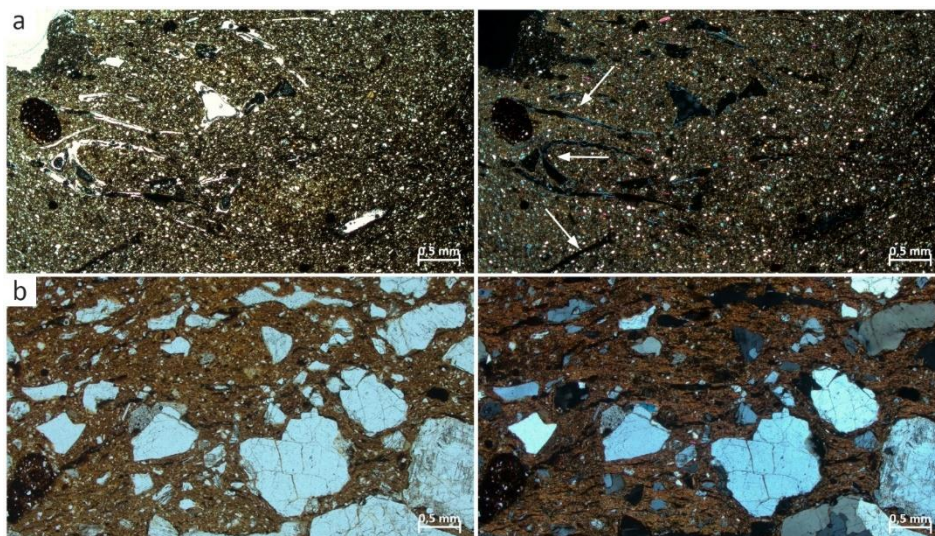


Figure 5. Thin section photomicrographs of pottery samples. (a) Chaff tempered sample 21 from the Jagodnjak-Krčevine site (Neral *et al.*, 2023), with arrows indicating voids resulting from the burned-out chaff and (b) sand tempered sample 291 from the Staro Čiče–Gradišće site (Neral *et al.*, 2024). Right microphotographs are taken in cross polarized light.

Copper Age

Recent studies on both early and late Copper Age pottery from eastern Croatia (Miloglav, 2016) and Hungary (Gucsi, 2021; Solnay *et al.*, 2023) reveal a common preference for grog as a tempering material. Grog has also been identified in late Copper Age pottery from Central Croatia (Neral *et al.*, 2024). Interestingly, however, grog was not the only temper observed. In addition to grog, mollusc shells were also identified, used either as the sole tempering material (Fig. 6a) or, less frequently, in combination with grog (Fig. 6b). These shells were likely collected from nearby rivers and watercourses, reflecting the use of locally available natural resources in ceramic production. Since shell tempering had not previously been recorded in the broader study region, this divergence in practice may point to a distinct technological approach developed by Copper Age communities in Central Croatia. While these communities relied primarily on local raw materials, their specific choice of temper sets them apart from neighbouring regions.

Mollusc shells are primarily composed of aragonite (which is unstable and readily transforms into calcite) or calcite itself. Their addition would have imparted properties similar to those provided by calcite temper. Because calcite has a thermal expansion rate similar to that of the ceramic matrix (Rye, 1976; Broekmans *et al.*, 2004; Shoval *et al.*, 2006), its inclusion can enhance vessel performance by reducing shrinkage, improving workability, increasing fracture

strength, and boosting resistance to thermal shock (Hoard *et al.*, 1995; Tite *et al.*, 2001). In addition, the platy structure of shell fragments contributes to the strength of the ceramic by helping to prevent crack propagation (Feather & Scott, 1989: 556).

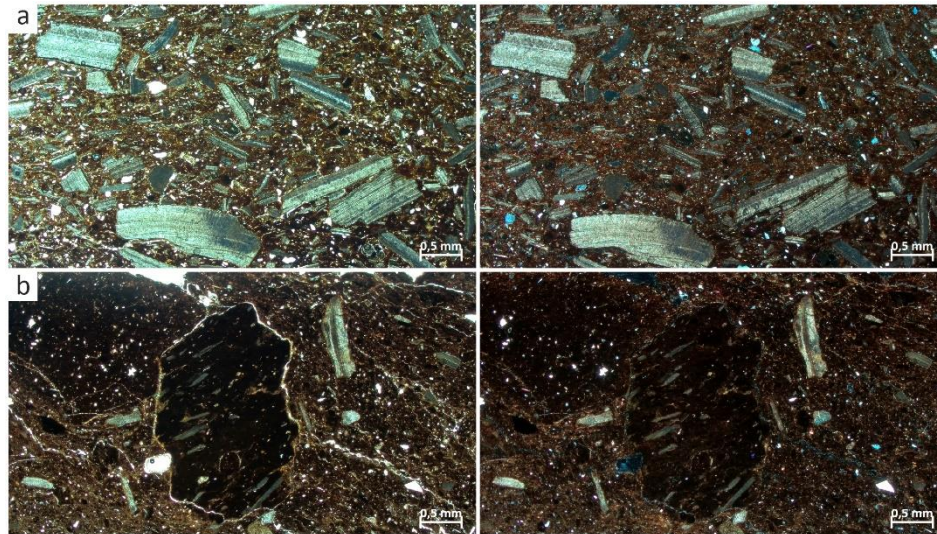


Figure 6. Thin section photomicrographs of pottery samples from the Staro Čiče-Gradišće site (Neral *et al.* 2024). (a) Sample 274, tempered with shell and (b) sample 276, tempered with both shell and grog, showing visible shell fragments within the grog. Right microphotographs are taken in cross polarized light.

Bronze Age

Following the Copper Age, the Bronze Age represents the next significant prehistorical period, characterized by the widespread presence of ceramic artifacts across archaeological sites in Croatia. Consequently, Bronze Age pottery was analysed in all case studies to develop a comprehensive understanding of technological choices and raw material use across different regions. A defining characteristic of Bronze Age pottery throughout Croatia is the use of grog (ceramoclasts) as a tempering material (Fig. 7a). This practice indicates a partial continuation or revival of the tradition of incorporating grog, which was previously observed during both the Neolithic period (Kreiter *et al.*, 2009, 2017) and the Copper Age (Miloglav, 2016; Forte, 2019; Gucsi, 2021; Solnay *et al.*, 2023). Grog is produced through circular technology, created by crushing fragments of previously fired ceramics, thereby ensuring a readily available supply of tempering material available in the settlement i.e., area of production. Previous studies on pottery production have shown that grog tempering was widespread among Bronze Age communities across the Pannonian Plain (Kreiter, 2007; Michelaki, 2008; Sofaer *et al.*, 2012; Žibrat Gašparič

& Dolenc, 2015; Kudelić *et al.*, 2018). Research highlights the functional benefits of grog tempering, particularly its role in enhancing the physical and mechanical properties of ceramic vessels. Given that grog is composed of fired clay, it has a thermal expansion rate very similar to that of the ceramic matrix, improving thermal shock resistance and fracture energy by reducing the development of microcracks that act as stress concentrators (Velde & Druc, 1999; Tite *et al.*, 2001). Experimental research (Neral *et al.*, 2025) similarly indicates that adding grog to the inclusion-poor clay used by Bronze Age communities in Central Croatia was essential to maintain the structural integrity of ceramics post-firing. The high concentration of swelling clay minerals in inclusion-poor clay increases water absorption, which, when converted into vapor during firing, creates internal pressure and leads to cracks. However, some grog-tempered ceramics contain only minimal amounts of grog (<15%), which has a negligible impact on the mechanical properties of the vessels. This characteristic, along with the presence of second-generation grog (grog within grog, Fig. 7b), may suggest that pottery recycling was a culturally significant practice. In cases where grog was derived from fragmented pottery, its reuse could reflect a tradition of maintaining ancestral connections and reinforcing cultural continuity (De Boer, 1974; Smith, 1989; Spindel, 1989; Chapman, 2000; Brück, 2006; Kreiter, 2007; Kudelić *et al.*, 2018; Quinn, 2022: 223).

Despite the widespread use of grog as a tempering material during the Bronze Age, variations in tempering preferences can be observed across different chronological phases. In Continental Croatia (including Central and Eastern Croatia), exclusive grog tempering is characteristic of the end of Middle and Late Bronze Age, while Early and beginning of Middle Bronze Age pottery is marked by the use of lithoclasts as tempering materials (Kudelić & Sirovica, 2022; Neral *et al.*, 2023; 2024). In contrast, Early Bronze Age pottery from Istria and central Dalmatia is mostly characterized by the exclusive use of grog tempering, while Middle and Late Bronze Age ceramics are predominantly tempered with calcite, either alone or combined with grog (Kudelić *et al.*, 2018, 2025; Neral *et al.*, 2023, 2024). The use of both tempering materials—grog and calcite—has also been documented in other regions, including the western Adriatic coast (Cannavò & Levi, 2018), central Dalmatia (Kudelić *et al.*, 2023), the broader Mediterranean (Albero Santacreu *et al.*, 2014), and Pannonia (Kreiter, 2007). Although the reason behind the addition of multiple non-plastic tempers has not been fully clarified, it may indicate that small quantities of grog may have been included for symbolic reasons—possibly to maintain a connection with ancestors or a place of origin (e.g., Gosselain, 1999; Kreiter, 2007; Larsson, 2009:

351–355; Pikirayi & Lindahl, 2013; Kudelić *et al.*, 2018; Holmqvist *et al.*, 2018; Quinn, 2022: 223)—while calcite was likely added for its functional properties.

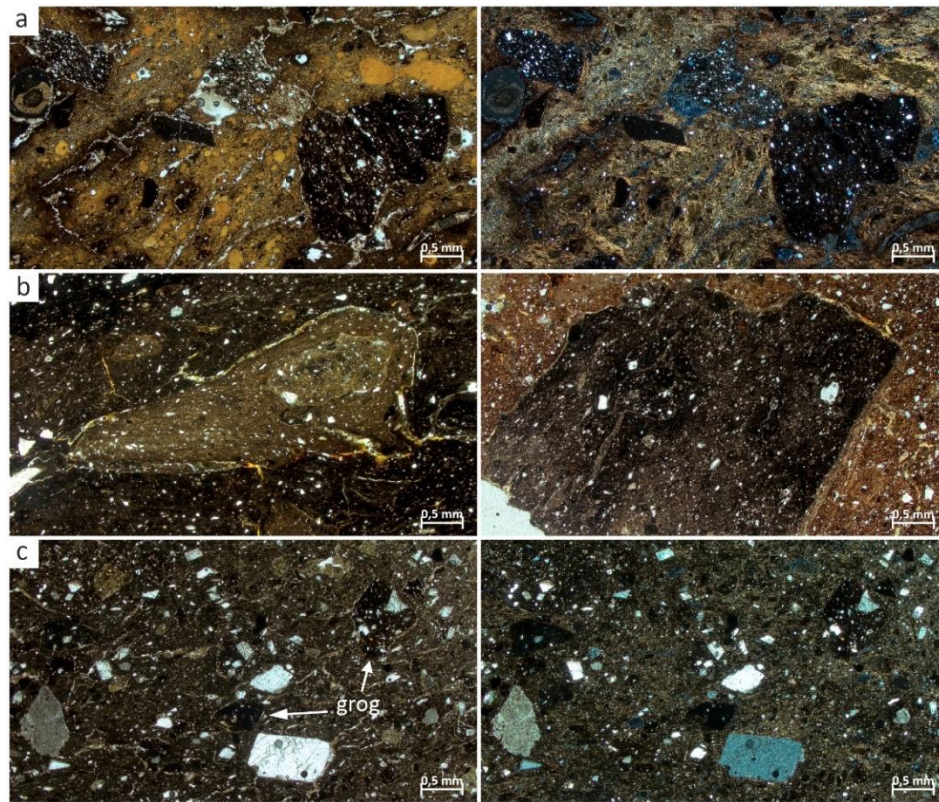


Figure 7. Thin section photomicrographs of pottery samples. (a) Grog tempered sample 190 from the Old Rakalj site (Kudelić & Neral, 2025), (b) second-generation grog fragments identified in samples 261 (left) and 262 (right) from the Staro Čiče–Gradišće site (Neral *et al.*, 2024), and (c) grog and calcite tempered sample 191 from the Old Rakalj site (Kudelić & Neral, 2025). Right microphotographs (a and c) are taken in cross polarized light.

Iron Age

By the end of the Bronze Age and into the Iron Age, the use of calcite as a temper material (Fig. 8a) along the Adriatic coast significantly increased and become the predominant choice (Kudelić *et al.*, 2023, 2024; Kudelić & Neral, 2025). This shift, marked by the complete abandonment of grog, suggesting a widespread change and stabilization in paste recipes.

In contrast, a distinctive tempering practice was observed for the Late Iron age pottery from Eastern Croatia, with graphite being the most commonly used temper (Fig. 8b). The widespread presence of graphite in Late Iron Age ceramics has been predominantly interpreted as a hallmark of Celtic cultural traditions (Waldhauser, 1992; Dizdar, 2001; Drnić & Mileusnić, 2012; Drnić &

Miletić Čakširan, 2014). However, previous research on graphite tempering also suggests functional advantages, such as enhanced resistance to thermal and mechanical stress, increased thermal conductivity, and improved resistance to chemical degradation (Kreiter *et al.*, 2013a and references therein). Given the rarity of graphite sources in the Pannonian Plain, it is generally assumed that such raw material was obtained through trade or exchange (Kreiter *et al.*, 2013a). However, in the case of the potters from Jagodnjak-Krčevine site, it is plausible that regional graphite was used, as the nearest source is located in the Papuk and Psunj mountains (Šinkovec & Krkalo, 1994; Drnić & Mileusnić, 2012), approximately 90 km from the settlement where pottery production is believed to have occurred.

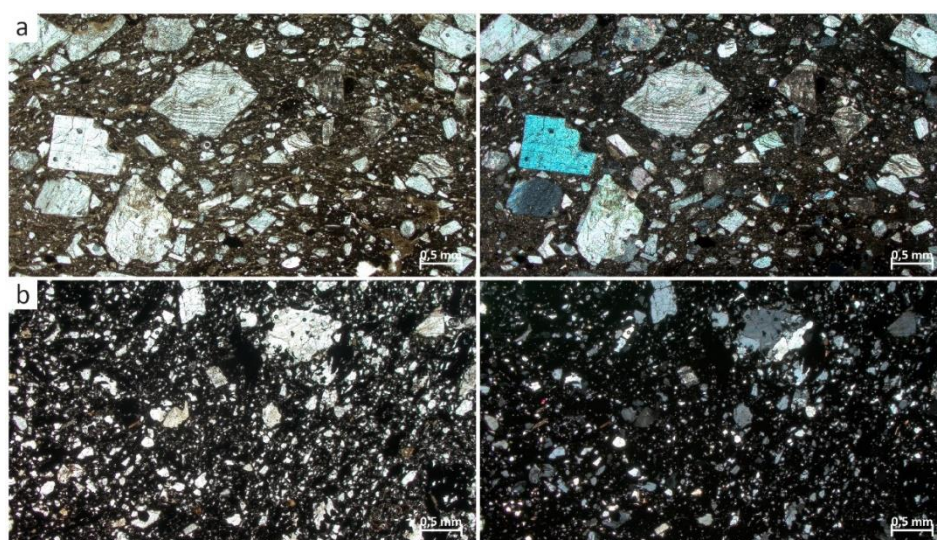


Figure 8. Thin section photomicrographs of pottery samples. (a) Calcite tempered sample 195 from the Old Rakalj site (Kudelić & Neral, 2025) and (b) graphite tempered sample 8991 from the Jagodnjak-Krčevine site (Neral *et al.* 2023). Right microphotographs are taken in cross polarized light.

Roman period

The Roman period marked significant transformations in ceramic production, characterized by a pronounced shift toward standardization that extended beyond the immediate needs of local communities (Peacock, 1982; Peña, 2007). This era saw the establishment of numerous pottery workshops dedicated to the mass production of ceramics for domestic use, storage, and transportation which led to the adoption of the potter's wheel, commonly known as the Roman wheel (Peña, 2007: 41). The expansion of communication and trade networks during this time

further increased the demand for ceramic vessels, which played a central role in the transportation of goods (Arthur, 2007: 164).

In response to these economic and infrastructural developments, ceramic workshops evolved into standardized production centres, functioning with the primary aim of optimizing output and minimizing production time. As a result, Roman-period ceramics are less reflective of individual households or communities and instead represent an industrialized mode of production. This shift toward specialization, although effective in fulfilling the demands of expansive trade networks, has led to the limited representation of Roman pottery within the scope of this research.

The analysis of ceramic samples from related studies of the Roman period in Istria highlights the use of a range of tempering materials. Potters in this region commonly employed grog, calcite, and sand sourced from the Adriatic Sea (Mange & Bezeczky, 2006; Sándor *et al.*, 2016). In contrast, this study has shown that in the continental regions of Croatia, sand was the predominant tempering material (Fig. 9; Neral *et al.*, 2023, 2024). These patterns reflect a deliberate reliance on locally accessible resources, situated within the broader framework of standardized ceramic production typical of the Roman period. The incorporation of tempering agents primarily fulfilled techno-functional roles, such as enhancing clay plasticity, reducing shrinkage during drying and firing, and improving resistance to thermal shock (Skibo *et al.*, 1989; Kilikoglou *et al.*, 1995, 1998; Hoard *et al.*, 1995; Vekinis & Kilikoglou, 1998; Neral *et al.*, 2025).

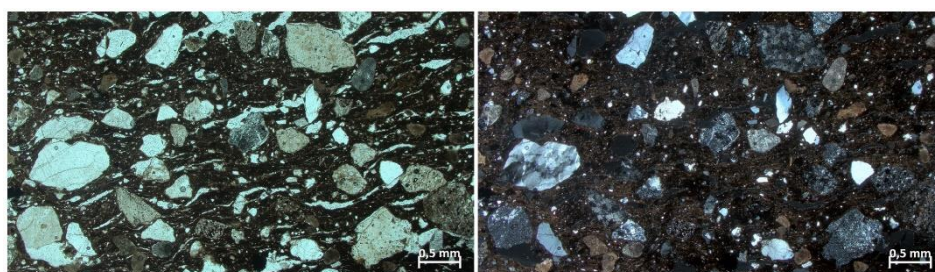


Figure 9. Thin section photomicrograph of sand tempered sample 256 from Staro Čiče-Gradišće site (Neral *et al.*, 2024). Right microphotograph is taken in cross polarized light.

Late Antiquity

Following the collapse of the Roman Empire, the expansive trade networks that had previously sustained large-scale ceramic production diminished significantly. This decline led to the gradual disappearance of numerous pottery workshops. As a consequence, ceramic manufacturing became

increasingly localized and less standardized, with production shifting toward smaller-scale practices that were executed with reduced precision (Arthur, 2007: 171–176).

In the northern Adriatic region, potters incorporated calcite into the clay matrix (Kudelić & Neral, 2025, Fig. 10), suggesting a departure from the standardized production practices typical of the Roman period and a closer alignment with the household-level ceramic production characteristic of the Iron Age. A similar tempering strategy is evident in the southeastern Alpine region (present-day Slovenia), where Late Antique pottery frequently contains intentionally added carbonate and calcite inclusions (Fazioli, 2012) a pattern consistent with recent findings from Podravina region (Kudelić *et al.*, submitted).

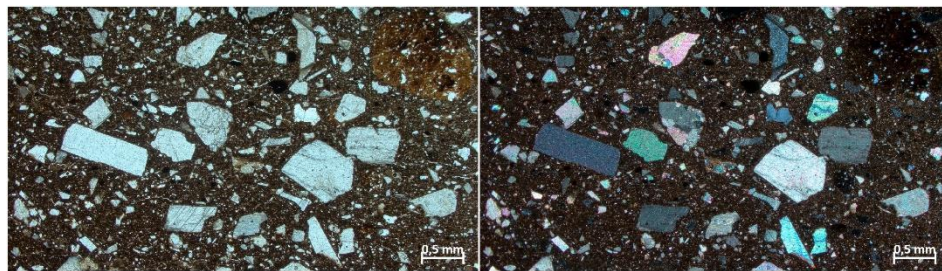


Figure 10. Thin section photomicrograph of calcite tempered sample 219 from Saint Theodore site (Kudelić & Neral, 2025). Right microphotograph is taken in cross polarized light.

Middle Ages

The trend toward increasingly localized and less standardized ceramic production, characteristic of Late Antiquity, continued into the medieval period (Arthur, 2007: 171–176). Nevertheless, the use of tempering materials remained a defining technological feature of pottery manufacture. Recent findings from the Podravina region indicate that the use of calcite as a temper persisted in the continental areas of Croatia until the 8th century (Kudelić *et al.*, submitted). From the 8th century onwards, broader socio-economic transformations (Bekić, 2016) were mirrored in changes to ceramic technology. During this period, lithoclasts and sand emerged as the predominant tempering materials which is also evident in High and Late Medieval pottery from Central (Neral *et al.*, 2024) and Eastern Croatia (Neral *et al.*, 2023).

Research has demonstrated that in Istria, potters continued to use calcite as a tempering material in Medieval period (Kudelić & Neral, 2025, Fig. 11b), reflecting a trend of utilizing suitable and locally available resources. Similar results have been reported in the southeastern Alpine region, where calcite and carbonate rocks were also employed as tempers (Fazioli, 2012).

Moreover, the morphological characteristics of calcite grains identified in medieval ceramics from Istria suggest that the material was likely sourced from cave environments, which are a prominent feature of the region's karstic geological landscape.

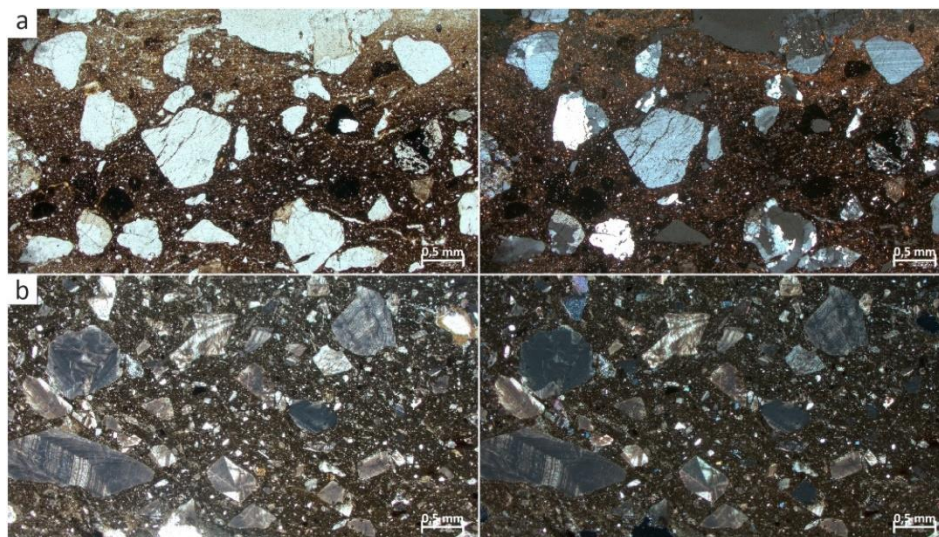


Figure 11. Thin section photomicrographs of (a) sand tempered, sample 8996 from Jagodnjak-Krčevine site (Neral *et al.*, 2023) and (b) sample 213 from Old Rakalj site (Kudelić & Neral, 2025) tempered with calcite of speleothem source. Right microphotographs are taken in cross polarized light.

3.4. Paste recipe preferences

The composition of ceramic paste used in vessel production, as demonstrated in the previous chapter, exhibited variability across different geographical areas, chronological phases, and cultural contexts. The present study reveals that the majority of analysed ceramics incorporated at least one type of tempering material. Specifically, among the assemblages examined within this doctoral research (Neral *et al.*, 2023, 2024; Kudelić & Neral, 2025), approximately 90% of the samples contained added temper. When the findings from related study is taken into account (Kudelić *et al.*, 2023), this percentage increases to an average of 93%, highlighting the consistent and deliberate practice of modifying clay to improve the functional properties of ceramic vessels. Notably, the types of tempering materials employed exhibit clear variation depending on the specific temporal and regional context (Fig. 12). A greater diversity of temper types was recorded in Continental Croatia (Fig. 12a) compared to coastal regions (Fig. 12b), reflecting not only distinct cultural traditions but also the crucial influence of environment in shaping technological decisions.

However, the inclusion of tempers appears to be related to vessel function. Ceramics intended for cooking, which would have been exposed to intense thermal fluctuations, commonly included tempering materials to enhance their resistance to heat stress and structural failure. In contrast, smaller vessels such as cups—presumably used for serving or storing rather than heating—were often manufactured without added temper (Neral *et al.*, 2023). This distinction supports the notion that prehistoric potters made informed technological choices based on the intended use of their products. These findings align with ethnoarchaeological studies (Arnold, 2000), reinforcing the interpretation that tempering practices were not arbitrary, but rather reflected a sophisticated understanding of material performance and functional requirements. These materials enhance clay workability, reduce shrinkage and plasticity, and increase thermal shock resistance and heating efficiency which has been corroborated by experimental studies (Skibo *et al.* 1989; Hoard *et al.*, 1995; Kilikoglou *et al.*, 1995; 1998; Hoard *et al.*, 1995; Vekinis & Kilikoglou, 1998; Hein *et al.*, 2008; Müller *et al.*, 2010; 2014; 2015). In this context, it is essential to acknowledge that the impact of tempers on the physical and mechanical properties of ceramics is also influenced by the type of clay to which they are added. Recent experimental study conducted as a part of this doctoral study (Neral *et al.* 2025) have demonstrated that incorporating tempers into sandy clay—the most commonly utilized clay type in the past—does not significantly influence the firing procedure or enhance the mechanical properties of ceramics, as measured by the point load index strength. Conversely, in inclusion-poor clay, the addition of tempering materials is essential for ensuring the structural integrity of ceramics after firing. The high concentration of swelling clay minerals in such clay increases water absorption, which, when converted into vapor during firing, generates internal pressure, leading to the formation of cracks. The incorporation of tempers such as grog, calcite, or sand mitigates this issue by reducing the proportion of swelling clays and absorbed water, thereby enhancing the ceramic's durability. Prehistoric potters appear to have been aware of these material properties, as all ceramic samples composed of inclusion-poor clay contain at least one type of temper, whereas those made from sandy clay occasionally lack added tempers. This suggests that potters adapted their technological processes to the specific properties of the clay they utilized. Clays with fewer natural inclusions required more intricate preparation techniques and extended firing durations but ultimately produced ceramics with greater mechanical strength, as evidenced by higher point load index strength values (Neral *et al.*, 2025). These findings align with recent research by Bernasconi *et al.*

(2024), further supporting the relationship between clay composition and the mechanical performance of ceramics. The majority of the analysed pottery contains between 20% and 30% temper. However, a smaller subset of samples exhibits only limited amounts of grog temper, typically up to 15%. Given that such a small quantity of grog is unlikely to have a significant impact on the vessel's physical properties, it is likely that this practice was driven by tradition. Potters may have deliberately incorporated small amounts of crushed ceramic, possibly as a symbolic gesture to preserve a connection with their ancestors or their place of origin, as suggested by some authors (Gosselain, 1999; Kreiter, 2007; Larsson, 2009: 351–355; Pikirayi & Lindahl, 2013; Kudelić *et al.*, 2018; Holmqvist *et al.*, 2018, Quinn, 2022: 223).

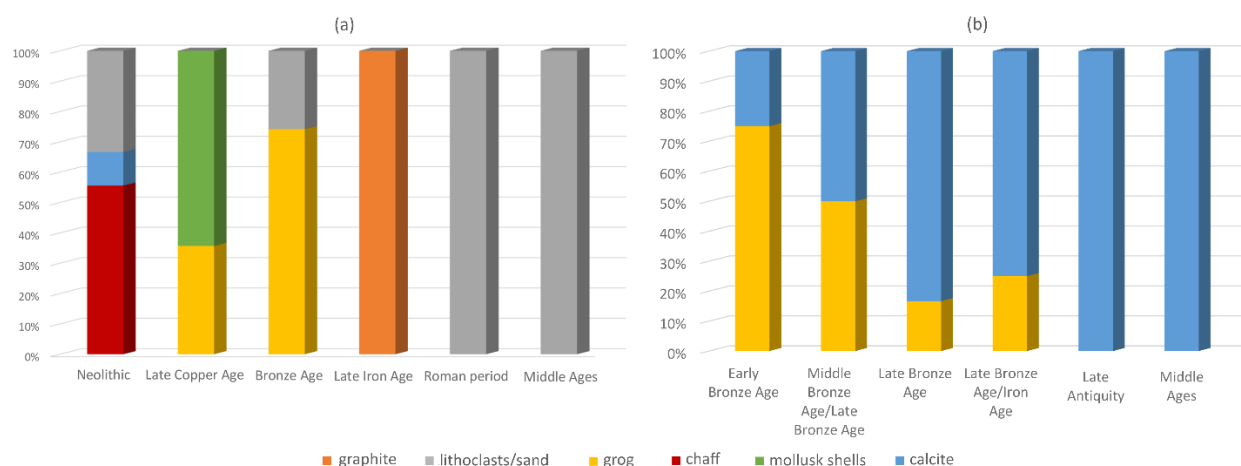


Figure 12. Proportion of pottery samples containing specific temper types across different historical periods in (a) continental and (b) coastal regions, based on data from Neral *et al.* (2023, 2024) for the continental regions, and Kudelić *et al.* (2023) and Kudelić & Neral (2025) for the coastal regions.

3.5. Variability of firing methods

The results indicate that most prehistoric pottery, especially those from the Neolithic, Copper and the Bronze age, regardless of their geographical origin, were fired at temperatures below 700 °C as indicated by the presence of 10 Å and 14 Å phyllosilicates (Cultrone *et al.*, 2001) (Fig. 13a). Ceramics were fired alongside fuel under conditions of incomplete oxidation, indicating uncontrolled firing processes, likely conducted in an open fire or shallow pit (Rice, 1987: 81, Kudelić & Sirovica 2022: 157–165; Neral *et al.*, 2023). These findings are consistent with previous ethnoarchaeological research on traditional firing techniques (Maniatis & Tite, 1981; Rye, 1981).

In the case of pottery from the Adriatic coast, this inference is primarily supported by the presence of calcite and dolomite (Fig. 13b), which begins to decompose at approximately 700 °C and is completely absent at temperatures exceeding 800 °C (Gruver, 1950; Cultrone *et al.*, 2001). Similarly, for ceramics from continental Croatia, a comparable upper firing temperature is suggested based on the medium-to-high optical activity of the matrix. Additionally, the presence of only 10Å phyllosilicates, corresponding to a dehydroxylated illite-like phase, further confirms firing temperatures in range from 700 to 800 °C (Gruver, 1950; Guggenheim *et al.*, 1987; Cultrone *et al.*, 2001; Rodriguez-Navarro *et al.*, 2009; Cultrone & Carrillo-Rosúa, 2020). The minimum firing temperature is estimated at 573°C, based on the presence of cracked quartz grains, indicative of quartz inversion (Velde & Druc, 1999: 128). However, there are exceptions to this general firing range. The sporadic occurrence of cristobalite (Neral *et al.*, 2023; Kudelić & Neral, 2025), a high-temperature polymorph of quartz, as well as spinel (Neral *et al.*, 2024), suggests firing temperatures exceeding 1000–1100°C (Cultrone *et al.*, 2001; Cultrone & Carrillo Rosúa, 2020) (Fig. 13c). Given the rarity of these high-temperature mineral phases, their presence is unlikely to indicate a distinct firing tradition but rather suggests secondary exposure to high temperatures, possibly due to accidental burning or exposure to high temperatures for some other reason. Such samples are typically fully oxidized and exhibit features consistent with the initial stages of vitrification.

From the Late Iron Age onward, kiln firing became the predominant method, resulting in higher firing temperatures and more controlled conditions. The increased occurrence of high-temperature mineral phases such as lime, spinel and cristobalite and occasional vitrification of the ceramic matrix in Late Antiquity and Medieval pottery from Central Croatia (Neral *et al.*, 2024) suggests that firing temperatures reached 800–1100 °C (Fig. 13d). This shift reflects a significant technological advancement, likely driven by the widespread adoption of kiln firing, improved control over firing conditions, and greater thermal efficiency. Such developments indicate a transition toward more standardized and specialized ceramic production, aligning with broader technological and economic transformations characteristic of these periods.

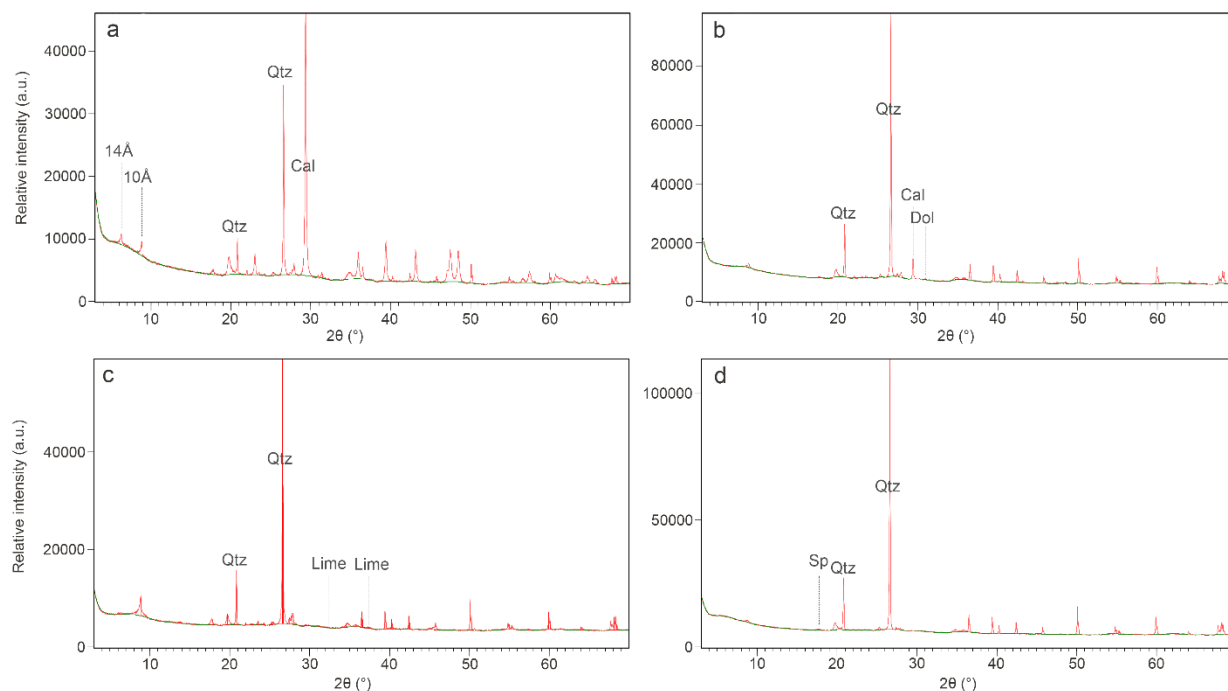


Figure 13. X-ray diffraction patterns of pottery samples indicative of different firing temperatures: (a) Copper Age sample 274 from Staro Čiče-Gradišće site, fired below 700 °C (Neral *et al.*, 2024); (b) Bronze Age sample 194 from old Rakalj site, fired below 800 °C (Kudelić & Neral, 2025); (c) Mediaeval sample 9108 from Jagodnjak-Krčevine site, fired at 800–900 °C (Neral *et al.*, 2023); and (d) Copper/Early Bronze Age sample 269 from Staro Čiče-Gradišće site, fired at temperatures occasionally exceeding 900–1000 °C (Neral *et al.*, 2024).

4. Conclusion

This doctoral research presents the first comprehensive, systematic study of pottery and its associated raw materials within the technological contexts of various historical periods and regions in Croatia. Employing a multidisciplinary analytical framework—including mineralogical, petrographic, geochemical, sedimentological, and mechanical methods—the study aimed to identify the types, selected physical and mechanical properties, and provenience of raw materials used by ancient communities. Additionally, it seeks to reconstruct technological aspects of ceramic manufacturing to enhance understanding of ancient production practices. By examining pottery from multi-period archaeological sites across Croatia and analysing potential raw materials collected nearby, several significant conclusions have been drawn:

1. Clay types: Two broad categories of clay types were identified: sandy clay and inclusion-poor clay. Sandy clay, characterized by moderate plasticity and shrinkage, was predominantly used due to its availability and ease of processing and firing. In contrast, inclusion-poor clay, with high plasticity and shrinkage, required more complex preparation, extended firing but yielded stronger ceramics. Its use, particularly among Bronze Age communities in central Croatia, suggests a strategic selection prioritizing material strength and durability over production efficiency.
2. Tempering materials: Various tempering materials were identified, including calcite, sand, lithoclasts, graphite, grog, chaff and mollusc fragments. In prehistoric periods, specific tempers correlate with certain timeframes, reflecting household or locally organized pottery production embedded within the community. In historical periods, a shift towards standardized production is evident, indicative of industrialized mode of production. Regional variations were also observed; for instance, continental Croatia exhibited greater diversity in added materials compared to Istria, where calcite predominated. These patterns underscore the environment's crucial role in shaping technological decisions, although certain choices cannot be fully explained solely by environmental factors and instead point to the influence of culturally embedded pottery traditions.
3. Provenience: The majority of the analysed ceramics appear to have been produced using locally sourced clayey materials, likely obtained within a 4 km radius of the production sites. This observation aligns with ethnographic studies indicating that potters traditionally procured raw materials in close proximity to their settlements, guided by principles of

minimal time investment and resource accessibility. However, instances of non-local clay usage have been identified, suggesting influences such as trade, migration, or other socio-economic dynamics during prehistoric and historical periods. Similarly, tempering materials were predominantly sourced locally, reflecting the availability of resources in the immediate environment. Nonetheless, the presence of non-local tempers in certain cases suggests that materials were obtained from more distant sources, possibly up to 90 kilometres away. This indicates that, in these instances, the selection of raw materials was likely guided by cultural considerations rather than solely by the accessibility of resources.

4. Paste preparation preferences: The application of tempering materials appears to have been functionally motivated. Cooking vessels, which were regularly exposed to high temperatures, typically contained tempers to enhance thermal shock resistance and improve heat distribution. In contrast, smaller vessels, such as cups likely used for serving or storage, were often produced without tempers. The study further reveals that the effectiveness of tempering materials was strongly dependent on the type of clay. In inclusion-poor clays, the addition of mineral tempers was crucial for maintaining structural integrity during and after firing. Sandy clays, by contrast, naturally retained stability without tempering materials. Moreover, inclusion-poor clays demanded more complex processing, including longer drying and firing times. These findings suggest that ancient potters adapted their technological process based on both the intended vessel function and the properties of the raw materials employed.
5. Firing techniques: Analysis of firing temperatures and atmospheres revealed distinctions between prehistoric and historical ceramics. Most prehistoric ceramics were fired below 800 °C under incomplete oxidation, indicating limited control over thermal parameters, consistent with open-fire or shallow pit-firing techniques. Exceptions with high-temperature mineral phases suggest occasional exposure to temperatures exceeding 1000 °C, possibly due to secondary factors like secondary fires. From the Early Iron Age onward, technological advancements, particularly the introduction of kilns, enabled higher and more consistent firing temperatures, along with better control of the firing atmosphere. This technological shift reflects improved firing processes and a transition towards more standardized and specialized ceramic production, corresponding with socio-economic transformations of the respective historical periods.

In conclusion, the doctoral research has successfully fulfilled all stated objectives, and all proposed hypotheses have been confirmed. The study represents a pioneering and systematic investigation of pottery production and raw material use, conducted through targeted, diachronic analyses of selected multi-period sites within a defined region of Croatia. Through the application of a multidisciplinary analytical approach, the research provides a nuanced understanding of ancient ceramic technologies, revealing how these practices developed and transformed across different periods and regions, and offering insights into the communities that created and employed them. Moreover, the study establishes a methodological framework for future research into ceramic production and raw material sourcing. Ultimately, the research underscores the importance of multidisciplinary collaboration, demonstrating that substantial, scientifically grounded insights can be achieved through the effective integration of diverse scientific disciplines and fields.

5. Literature

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6. Biography of the author

Natali Neral was born in 1996 in Rijeka, Croatia. She completed her secondary education at the gymnasium in Rijeka and enrolled in the undergraduate program in Geology at the Faculty of Science, University of Zagreb, in 2015. She earned her bachelor's degree in 2018 and went on to complete the graduate program in Geology and Paleontology at the same institution, graduating with honours (*cum laude*) in 2020. In 2021, she joined the Institute of Archaeology in Zagreb as a research assistant on the interdisciplinary project Technological Features and Cultural Practices in Prehistoric Pottery Traditions in Croatia (UIP-2020-02-3637, acronym: prePOT), funded by the Croatian Science Foundation. Within the scope of this project, she has specialized in the study of raw materials used in pottery production, with a particular focus on mineralogical, petrographic, and geochemical analyses. She is currently pursuing her doctoral studies at the Faculty of Mining, Geology, and Petroleum Engineering, University of Zagreb.

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