Production and function of Neolithic black-painted pottery from Schela Cladovei (Iron Gates, Romania)

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Abstract: *This paper presents for the first time the results of a combination of petrographic, geochemical and organic residue analyses of early Neolithic ceramics from the Iron Gates region of the Danube basin. Eleven early Neolithic potsherds from Schela Cladovei (Romania) were analysed in detail. The results of the petrographic analysis show that the ceramics were made with the same recipe that was used by* Starčevo-Körös-Criș *potters elsewhere in southeastern Europe. The SEM-EDX analysis shows one of the earliest uses of Mn-rich black pigments to decorate Neolithic European ceramics. Organic residue analyses detected dairy, non-ruminant and ruminant adipose fats. No evidence of aquatic resources was detected. In summary, the early Neolithic potters at the Iron Gates, although able to make coarse and more sophisticated painted ceramics, did not make specific vessels for a specific use.*

Keywords: early Neolithic, polarised microscopy, SEM-EDX, GC-MS, GC-c-IRMS, ceramic technology, pottery paint

Highlights

- This is the first time that the same Neolithic sherds were analysed by archaeometric and organic residue analyses, to understand whether there is a correlation between the paste recipe and the function of the pot
- The Starčevo recipe for pottery production at the Iron Gates is compared with that of the Starčevo-Körös-Criș ceramics from other sites in Romania, Serbia and eastern Croatia
- Organic residue analyses show a range of culinary functions, based on terrestrial food sources
- This is also the first time that SEM BSE images and EDX analyses are used to investigate the black paint coatings of early Neolithic pots from the Balkans

Introduction

The earliest Neolithic of the central and northern Balkans is represented by the Starčevo-Körös-Criș (SKC) culture complex (Fig. 1). Most SKC sites lie north of the Balkan Mountain chain (Stara Planina). The terminology used tends to follow modern political divisions – 'Starčevo culture' in Serbia and northern Croatia, 'Criș culture' in Romania, and 'Körös culture' in Hungary. Radiocarbon dates for settlements across the entire SKC geographical range span the period from c. 6200–5500 cal BC though few, if any, sites north of the Danube have dates earlier than c. 6000 cal BC. Potsherds are the most commonly recovered artefacts from Starčevo sites, and perceived changes in vessel form and decoration have been used to subdivide the SKC time-range into 'phases'. Various regional typological sequences have been proposed – e.g. Aranđelović-Garašanin (1954), Dimitrijević (1974; 1979), Lazarovici (1979; 1984). These chronological schemes are widely used in Serbia, Croatia and Romania, respectively.

In this paper we report on archaeometric analyses of a series of SKC sherds from Schela Cladovei in southwest Romania. These are the first archaeometric analyses of early Neolithic pottery production from a site in the Iron Gates region, and the first combined analyses of pottery production and function at any SKC site.

Schela Cladovei (44°37'46.36"N, 22°36'0.34"E) is a large late Mesolithic and early Neolithic settlement on the left bank of the River Danube in the area known as the Iron Gates (Fig. 1 inset). Remains of SKC culture settlement at Schela Cladovei extend for at least a kilometre along the bank of the Danube, and at least 200m back from the river bank (Boroneanț et al. 1999). Parts of the site were excavated by the late Vasile Boroneanț between 1965 and 1991. Since 1992 the excavations have been conducted as a joint Romanian-British research project, initially co-directed by V. Boroneanț and C. Bonsall and latterly (2007-onwards) by A. Boroneanț and C. Bonsall.

The 1992–1996 excavations focused on Area VI, covering c. 55m^2 immediately adjacent to the Danube (Fig. 2). Early Neolithic finds from this area included nine pits and pit complexes, thousands of pottery sherds, hundreds of chipped stone artefacts (of flint, obsidian and quartz/quartzite), ground stone and bone artefacts, as well as large numbers of mammalian and fish bones and shells of freshwater molluscs reflecting an economy that was based on a combination of farming, fishing and foraging – a pattern that appears to have been characteristic of SKC culture settlements throughout the Iron Gates region. Pottery was recovered from all nine pit features, as well as from the 'cultural layer' between the pits. All the sherds discussed in this paper come from Area VI. Subsequent work at Schela Cladovei has focused on Area VII, located c. 20m from Area VI (Fig. 1) and the river bank. Organic residue analyses have been performed on sherds from several early Neolithic pit features in this area (Cramp et al. 2019).

[Figure 1 here]

The chronology of the SKC culture settlement at Schela Cladovei currently rests on 10 single-entity AMS ^{14}C dates on artefacts made from terrestrial animal bone, all recovered in the 1992–1996 excavation of Area VI (Bonsall 2008). The dates range between c. 6000 and 5600 cal BC. Compared to ^{14}C evidence from early Neolithic sites elsewhere in Romania (cf. Biagi et al. 2005; Mirea 2011), this broad date range suggests more than one 'phase' of the SKC culture could be represented by the finds from Area VI at Schela Cladovei. However, the dates currently available are too few and unevenly distributed to establish a coherent absolute or relative chronology for the various pit features in Area VI.

[Figure 2 here]

The pottery from the 1992–1996 excavations at Schela Cladovei showed all of the typical traits of a SKC domestic assemblage (Chapman forthcoming). The material was divided into four series – dishes, bases, neckless bowls/jars and necked bowls/jars - with vessel sizes based upon rim diameters ranging from 5cm to over 45cm (Fig. 2 bottom). Vessels were decorated by polishing the leather-dry pottery surface with a tool before firing, slipping (coating, usually before the pot is completely dry, with a homogenous suspension of clay in water, often coloured with added oxides), painting and/or incised patterns, but others were undecorated. The black-on-red painted decoration was typical of late SKC painting throughout the region (for the Banat, Lazarovici 1979; for the Alföld, Oross 2007; for Slavonia, Dimitrijević 1974; for Moldavia, Ursulescu 1984).

Sampling and scientific techniques

The total excavated sample in Area VI amounted to over 7,000 sherds in an area of 52.5m^2 (by volume, c. 84m^3) (Chapman, in press). Eleven potsherds with a range of surface treatments and decoration, such as black-on-red painted motifs, red-slipped and red-polished, incised patterns, and plain undecorated ware were selected (Table 1; Fig. 2 top). Dark brown/black painted decorations include curvilinear geometric (SCL6, 9 [a rim sherd of an open dish]), spirals (SCL5), parallel linear vertical bands (SCL4), and dark painted vertical convergent(?) bands (SCL11). A ceramic fragment (SCL2) had faint traces of possible red-ochre on the surface. Although small, the sample of 11 sherds represented all of the main visually identified fabric types. Only SCL9 was from a sufficiently large sherd that the shape of the vessel could be determined with certainty.

All samples were prepared as polished thin sections without cover slips, and were analysed using both a Zeiss Axiophot polarised light microscope and a Hitachi S-3700N variable pressure (VP) scanning electron microscope fitted with an Oxford Instruments AZtec energy-dispersive X-ray spectrometer (SEM-EDX).

Polarised microscopy analyses of thin sections allow the recognition of certain steps of ceramic chaîne opératoire, such as clay provenance and processing (e.g. removal or deliberate addition of inclusions, called 'temper'), surface treatment (polishing, slipping, painting), and firing (see Spataro 2017). High magnification backscattered electron (BSE) SEM images allow the study of the sample microstructure (ceramic fabrics, slips and painted coatings, and firing temperature), and SEM-EDX analyses measure the chemical composition (concentrations of major and minor oxides) of samples. Four bulk analyses of areas c. 1.4 × 1.0 mm were carried out on each sample. Nine elements (Na, Mg, Al, Si, K, Ca, Ti, Mn, Fe) were quantified[1]; the results were converted into oxide percentages, which were normalised (oxygen by stoichiometry) (e.g. Spataro et al. 2015, 177–183). Principal component analysis (PCA) was used to help interpret the SEM-EDX results. PCA of the correlation matrix was performed using PAST v.3.18 (Hammer et al. 2001). All principal components were examined. EDX compositional maps were used to examine the elemental concentrations on painted layers, interlayers (polished or slipped) and the body paste of three samples (SCL4, 5 and 11)[2].

All sherds were also sampled for organic residue analyses. Well-established protocols of extraction and methylation of lipids were applied in the organic residue analysis (Craig et al. 2013; Correa-Ascensio and Evershed 2014). The extraction protocol requires the addition of methanol (4mL) to 1g of pottery powder and the mixture was ultrasonicated for 15 min. Sulphuric acid (H_2SO_4, 800µL) was used to acidify the suspension, which was then heated for 4 hours at 70ºC. Finally, lipids were extracted from centrifuged pottery powder with *n*-hexane (3x4 mL). An internal standard (10 µL of hexantriacontane $C_{36:0}$) was added to all the samples to quantify the relative abundance of lipids. The samples were then analysed directly by Gas Chromatography-Mass Spectrometry (GC-MS) and Gas Chromatography-Combustion-Isotope Ratio Mass Spectrometry (GC-c-IRMS). All samples were also extracted using 2:1 DCM:MeOH (3x2mL) to produce a total lipid extract (TLE) following established protocols (Evershed et al. 1990, Correa-Ascencio and Evershed 2014). The extracts were then dried under N_2 and derivatised with N,O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) heated at 70ºC for 1 hour. An internal standard (10 µL of hexantriacontane $C_{36:0}$) was added to all the samples. The derivatised extracts were then analysed by High Temperature Gas Chromatography-Mass Spectrometry.

Results

Thin section analyses

Eleven samples were analysed and three fabric groups were defined. All groups were non-calcareous and had been tempered (=deliberately added) with plant matter, as abundant planar and oriented voids were recognised, and leaves and possible wheat glume bases were detected.

Group 1 (SCL1, 7) is a typical SKC fabric, with a sandwich-core, which is due to firing in an oxidising atmosphere, but with insufficient oxygen available at the centre to completely burn out the plant matter added as temper (Fig. 3a). The fabric is micaceous, rich in fine quartz silt, with some scattered coarser inclusions of metamorphic and (igneous?) rock fragments, and sub-rounded to rounded polycrystalline limestone fragments of different sizes. Five subgroups, all of which are also chaff-tempered, were distinguished. Group 1 *subgroup a* (SCL3) is like Group 1 with occasional metamorphic rock fragments, but with fewer and finer limestone inclusions. Group 1 *subgroup b* (SCL8) contains fine very well-sorted quartz inclusions with fewer scattered coarser inclusions than Group 1 samples (Fig. 3b). *Subgroup*

[1] Phosphorus and sulphur were detected and measured in the pigments.
[2] The carbon peak can be identified by EDX – and it is clearly absent in elemental maps of the paint, which are extremely accurate and precise (see also Fig. 7f).

c (SCL10) is similar to *subgroup b* but with more abundant limestone fragments and fewer quartz inclusions, and no other rock fragments. *Subgroup d* (SCL11) contains more abundant quartz inclusions (slightly coarser than in Group 1) than the other subgroups, and scattered granitic rock fragments, which are finer than in Group 1. *Subgroup e* (SCL2) is slightly more micaceous, and it contains finer and less abundant silt than the other fabrics in Group 1, but with some limestone fragments.

Group 2 (SCL4, 6) was probably tempered with both plant material and sand. Group 2 contains less plant matter than Group 1 and subgroups. The sand includes granitic rock fragments and some metamorphic rocks (Fig. 3c). Although it can be difficult to determine by petrography whether sand was naturally present or added deliberately to the clay, the rock fragments, which are seldom present in SKC pottery (Kreiter and Szakmány 2011; Spataro in press), provide a strong indication that the sand in Group 2 was deliberately added. Group 2 *subgroup a* (SCL9) was tempered with plant matter and sand, and its sand includes, as well as granitic rock fragments, more abundant high-grade metamorphic rock fragments than Group 2.

Group 3 (SCL5) is very fine and has fewer limestone fragments than the other groups. It was chaff-tempered (Fig. 3d).

All fabric groups contain fine alluvial silt and some have more or less frequent sub-rounded limestone, granitic and metamorphic rock fragments in different percentages. All groups contain rather thick lamellae of biotite, which are likely to come from granitic rocks. The sand of Groups 1 and 3 is similar and it might come from the same clay basin, as could the finer sand in Group 2. However, the sand used to temper Group 2 *subgroup a* has a higher content of high-grade metamorphic rocks, as well as granitic fragments.

A variety of typical SKC surface treatments were applied to vessels, which in some cases were made using the same fabric (see Group 1 and subgroups with no decoration [some red-coated], with incised decoration and with painted decoration). Painted pots were made using all fabric types; both chaff- (Groups 1 and 3) and chaff-and-sand tempered (Groups 2 and 2 *subgroup a*) pastes were used.

Black-on-red ceramics were painted on either polished or slipped surfaces. There is no correlation between the choice of slipping or polishing and a specific fabric type (Table 1).

[Figure 3 here]

[Table 1 here]

SEM-EDX analyses

All pots were well-fired, as the BSE images show good sintering of the clay but not vitrification (see Fig. 4a), but some charred plant remains survive (Fig. 4b). Only one sample (SCL8) revealed initial vitrification of the clay filaments at the surface.

The EDX results show strong similarities in chemical compositions between sherds; in particular, magnesium (MgO, 1.4–1.9%), potassium (K_2O, 2.0–2.3%), titanium (TiO, 0.8–1.0%) and iron (FeO, 5.3–6.4%) oxides contents are consistent (Table 2). However, sodium (Na_2O, 1.1–1.7%) and calcium (CaO, 3.4–6.1%) are more variable, mainly due to differences in the abundance of silt (including variable percentages of albite) and limestone fragments.

In PCA Fig. 5, samples SCL1 and 7 cluster apart from the other Group 1 samples, as they are richer in calcium oxide, due to the coarser and more abundant limestone fragments. The other samples show some heterogeneity within and between groups.

[Figure 4 here]

[Figure 5 here]

[Table 2 here]

Slipped and polished surfaces

Macroscopically (with the naked eye) and microscopically (with the polarised light microscope), slipped and polished surfaces are difficult to distinguish: in some instances only chemical analyses help to distinguish between polished and slipped layers, on the basis of the compositional similarities/dissimilarities of the interlayer between the paint and the body paste of the sherds (see Spataro 2016).

Three sherds were polished and painted (samples SCL6, 9 and 11), three samples were slipped (SCL2, 4, 5), and two of these (SCL4 and 5) were also painted. It is not possible to determine whether the plain sherds were from partially decorated vessels.

Red slips were analysed by SEM-EDX (Table 3). Slipped layers are rich in iron oxides, alumina and silica, with occasional fine inclusions of muscovite and quartz. This suggests that slips were made of very fine levigated (depurated) clays, to which iron oxides were added and mixed with water (see Figs 4c and d; Spataro 2016). One sample (SCL4) had both interior and exterior surfaces slipped. Both surfaces were analysed, showing that the same pigment was used (see Table 3).

Black paint

In four of the five pots decorated with black paint (sample SCL4, 5, 6, 9 and 11) (Figs. 6 and7a), the paint was visible in thin section and it could be analysed with bulk analyses at different magnification by SEM-EDX (e.g. Figs. 7b and 8h; Table 3; the thin section of sample SCL4 did not include any paint). The magnification of the analyses varied depending on the thickness of paint visible on the thin section.

BSE SEM images show a painted coating with a mixture of inclusions, including abundant nodules, which the EDX results show are rich in manganese (>50%) and iron (>20%) oxides (e.g. sample SCL9), or iron-rich grains (e.g. SCL5). In some cases, quartz grains are also visible (Fig. 8c). The composition and the microstructure of the painted layers suggest that metal oxides (oxidised manganese dioxide) were finely ground and probably mixed with clay and water.

Occasionally, the painted layers have some voids, which might be due to the drying of the paint or to the fact that two coatings might have been applied to the surface. Although the paint appears to have been fired, as it is compact and sintered (Fig. 9a and b), there is no evidence that the pot was fired twice, once before the painting and once after. Thus, the paint was probably applied before firing.

[Table 3 here]

EDX elemental mapping

To visualise the elemental distribution within body fabrics, slips and black painted layers, EDX elemental composition maps were run on samples SCL4, 5, 6 and 11 (Figs. 7 and 8). The maps confirm the presence of slips with high concentrations of iron (see Fig. 7d) and potassium and low calcium contents, and the high iron and manganese contents in the black pigment, in contrast with the low iron and absence of manganese in the body paste (Figs. 7e and 8e). The black paint has a relatively low concentration of aluminium, silicon and magnesium (Figure 8b, c, and g; Table 3). The presence of quartz grains in the painted layer is visible in Figure 8a and c.

[Figure 7 here]

[Figure 8 here]

[Figure 9 here]

Organic residue analyses

Potsherds from Schela Cladovei were previously investigated by organic residue analyses (Craig et al. 2005; Cramp et al. 2019). New organic residue analyses have been carried out on 11 samples, also analysed with other scientific approaches (Table 1) to have all the mineralogical, chemical (inorganic and organic) and isotopic information of the vessels.

Sherds yielding lipid residues are summarised in Table 4. All sherds analysed from Schela Cladovei contained a concentration >5 $\mu g\ g^{-1}$ and show significant quantities of saturated and unsaturated fatty acids, *n*-alkanes, *n*-alcohols and dicarboxylic acids (samples SCL 1, 2, 8) (Table 4). In contrast with other studies from the same site (Cramp et al. 2019), no aquatic biomarkers have been identified.

GC-c-IRMs analysis of palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) fatty acids indicates that molecular composition derives from different food sources (Fig. 10). Most of the $\Delta^{13}C$ ($\delta^{13}C_{18:0}$-$\delta^{13}C_{16:0}$) values are consistent with the references available in Europe for the ruminant adipose fats[3] (n=6), whose $\Delta^{13}C$ ranges from -2.61 to -0.55. Two extracts can be related with the processing of mixed ruminant and non-ruminant adipose fats. Three samples reflect the processing of dairy products (Table 4; Fig. 10) that has been previously reported at the site (Craig et al. 2005).

[Table 4 here]

[Figure 10 here]

Biomarkers characteristic of aquatic resources, such as *ω*-(o-Alkylphenyl)alkanoic acids (APAAs) (e. g. Hansel *et al*. 2004, Evershed *et al*. 2008), or cereals, such as alkylresorcinols (Hamman and Cramp 2018; Colonese et al. 2017), have not been reported in our samples. However, aquatic biomarkers have been recently identified in pottery sherds from the site highlighting a high diversity in pottery use.

[Table 5 here]

The combination of different analytical techniques has allowed us to explore the relation between manufacture techniques (fabric groups) and main functionality of the pots sampled. Chi-square test reflects an independence between fabric and $\Delta^{13}C$ (Chi-square = 22; gl = 20; p-value = 0.341) (Table 5). Due to the fragmentary nature of the pottery, it is not possible to detect, or exclude, an association between vessel shape and function.

Discussion

The ceramic chaîne opératoire at Schela Cladovei was based on the same recipe used by SKC potters elsewhere. The potters collected non-calcareous and micaceous-very micaceous clay and temper from sources derived from limestone, meta-granite, metamorphic rock fragments, which are found locally and regionally. The site is located in an area of extensive alluvial deposits composed of unconsolidated gravels sand, silt and clay, and loessic deposits, although there are outcrops of sandstone and flysh c. 5km to the northwest (Petrescu and Ciobanu 1966). Before reaching the site, the Danube passes through mica schist and granitic outcrops, limestone and conglomerates and dolerite (Răileanu et al. 1968). During the Schela Cladovei excavations, it was noted that a small percentage of SKC sherds were tempered

[3] Data obtained on ruminant, non-ruminant, dairy and marine resources throughout Europe have been compiled (Dudd 1999; Spangenberg et al. 2006; Bell et al. 2007; Craig et al. 2011; Spiteri 2012; Recio et al. 2013; Cramp et al. 2014; Carrer et al. 2016) to compare isotopic values of the main fatty acids.

with iron pyrite, which was believed to have come from a source within the Iron Gates Gorge. These sherds have not been analysed archaeometrically, however, and the precise location of the source(s) is unknown. As at other SKC sites, the potters did not process the clays carefully, as clay pellets, poorly-mixed and occasional fine bone fragments are present in the pastes. The clays were then tempered with cereal chaff, and probably in one case, with chaff and sand.

The SKC communities in the Iron Gates, at Schela Cladovei and at Lepenski Vir (see Perić and Nikolić 2004, 181) mainly used alluvial clays, heavily tempered with chaff, to make their vessels, like SKC potters throughout Romania, Serbia and Slavonia (e.g. Spataro 2011a; 2011b; 2017) and in Hungary (see Kreiter *et al.* 2013, 137). The combination of chaff and sand temper, recorded in one of the Schela Cladovei pots, has been detected at several sites, in particular in Romania (Spataro in press). As at other SKC sites, plant temper at Schela Cladovei was not very finely cut (compared to some of the few Vinča chaff-tempered fabrics from Romanian Banat: Spataro 2014; see Fig. 3b) and the size and abundance of plant temper seems to be unrelated to whether the clay was also tempered with sand (plant-and-sand tempered fabrics have a variable quantity of plant material, generally similar to that present in the plant-tempered only fabrics).

At Schela Cladovei, some ceramics were finely polished, in order to obtain a smooth surface on which the paint was applied. Others were slipped, as slipping would have facilitated the process of surface polishing, to obtain the same shiny effect (J. Kneisel, pers. comm. 2017). Slips were prepared using the same procedure and formula employed to prepare slips for monochrome and white-on-red painted ceramics from the earliest SKC phases elsewhere (Spataro 2016). Seventy-seven SKC red-slipped pots (including white-on-red and painted on surface) from 12 sites were analysed (Spataro 2016, table 1), showing that the same knowledge of how to make iron-rich clay slip coatings transmitted over a wide geographical area, spanning from Slavonia through Serbia and eastern Transylvania. The slips are very fine, uniform layers 0.15–0.20mm thick, composed mainly of alumina and silica (c. 70%), mixed with iron oxide (10–25%) and a small content of manganese oxide, which is naturally present in ochre (Spataro 2016, 170).

Parallel linear black-painted ceramics are found throughout the SKC distribution, as far north as Slavonia (e.g. Zadubravlije and Galovo, Minichreiter 1992, 70; 2007, 97) and Hungary (e.g. Alsónyék-Bátaszék, Bánffy et al. 2010, figs. 11 and 12). Nevertheless, there has been no archaeometric analysis of black pigment, except at the site of Sălcuţa in Oltenia, where pyrolusite was detected in the one sherd of which the black paint was analysed (B. Constantinescu, pers. comm. 2018; Constantinescu et al. 2007, 285, 287).

All the black paint analysed at Schela Cladovei contains high concentrations of Mn and Fe derived from earths that were rich in these elements, probably mixed with fine clay (generally alumina and silica are less than 50%) and water. This is not the only known case among Starčevo black painted ceramics (Spataro in press). Ferromanganese and manganese oxides are stable in all firing conditions (Ellis 1984, 119), and were therefore ideal for the SKC potters, as the pots were probably fired in bonfires. In Romania, abundant pyrolusite and jacobsite can be found in the eastern Carpathians (Vatra Dornei-Iacobeni region); traces of pyrolusite, mixed in particular with limonite, are found in the rest of Romania (B. Constantinescu, pers. comm. 2018; Rădulescu and Dimitrescu 1966). There may have been a source of ferromanganese and manganese oxides close to the site, but this needs to be investigated further.

Manganese-rich ores were also used for the black paint of the Neolithic ceramics at Dimini, in Greece (analysed by X-ray fluorescence spectroscopy [XRF]; see Papadopoulou et al. 2007). Angeli et al. (2018, 179) used XRF analyses and Raman spectroscopy to argue that the common form of Mn-rich mineral in black-painted middle Neolithic Apulian *figulina* ware (5000–4500 BC) is pyrolusite, which converts during firing at temperatures above 450°C into bixbyite "and then combines with the iron oxide to form the manganese-iron oxide jacobsite above 900°C". On the other hand, the black-painted decoration used on early Neolithic Lagnano da Piede-style Apulian ceramics, analysed by Raman and Laser Induced Breakdown Spectroscopy, was obtained using carbon black (Angeli et al. 2018, 174).

In the Eneolithic Cucuteni culture, which flourished east of the Carpathians between *c.* 4800 and 3000 cal BC (Monah and Monah 1997), ceramics were profusely painted with red, white and brownish-black, respectively using hematite, calcite and manganese oxides (birnesite and manganite which were transformed into bixbyite; Ellis 1980, 211–230; see also Bugoi et al. 2008); pyrolusite and/or jacobsite were also detected by XRD, Raman, FT-IR and Atomic Absorption Spectrometry (see Buzgar et al. 2013).

Manganese oxide was used as a paint in Palaeolithic rock art, e.g. at the caves of Lascaux in France and Ekain in Spain (see Chalmin et al. 2003), but its use by the SKC potters seems to be the earliest proved in European ceramics. SEM-EDX analyses of 'dark painted' pottery from the early Neolithic Bulgarian site of Dzhulynitsa (Dzhanfezova et al. 2014, 149) did not detect elevated Mn concentrations on black surfaces, and the authors suggest that the colour might be due to different oxidation conditions, possibly obtained by the application of water-insoluble organics material on the surfaces[4]. In Macedonia and Thrace, manganese and iron-manganese pigments appear from the late Neolithic onwards (Yiouni 2001, 10). In eastern Macedonia, pottery decorated with graphite (a crystalline form of carbon) is also common in the same period (op. cit. 25). At Schela Cladovei, firing temperature and conditions for plain, impressed, incised, red-slipped and painted ceramics were similar; the ceramics were fired in an oxidising atmosphere and at relatively low temperatures, generally not exceeding 650–700°C, as suggested by the sintered but non-vitrified fabrics and the abundance of charred remains. These firing conditions could have been obtained in bonfires; kilns were not required (Livingstone Smith 2001, 999). There is little evidence of kilns in SKC settlements (see Minichreiter 2004; Nica 1977; Tencariu 2015).

No correlation between ceramic typology and fabric was detected, as the same recipe was used to make vessels with different surface treatments and decorations, including plain, incised, slipped, polished and painted ware. The slipped pot (SCL2) and one of the black-

[4] The authors also suggest that possible graphite or charcoal may have been applied in some cases after firing (Dzhanfezova et al. 2014, 149).

painted ceramics (SCL5) were made with similar but finer fabrics than the other painted pots, whereas other polished or slipped and painted ware (SCL4, 6, 9 and 11) was made with pastes with coarser inclusions. Both plant and plant-and-sand tempers were used to make the sophisticated painted ceramics. Painted ceramics were made with fine and relatively coarse fabrics (e.g. SCL5 and SCL11), adding chaff or chaff-and-sand temper.

The use of the same recipe to make both painted and undecorated ceramics, which is a typical trait of the SKC culture over a wide territory, from the Iron Gates to Slavonia and through the Banat Plain and Transylvania demonstrates that the pottery production was not isolated from the SKC society (e.g. Spataro 2011b; 2016). Archaeometric analyses confirm the initial impression that strong similarities at the level of shape series, shape types and decorative styles can be detected in coeval sites and groups of sites at distances up to 200km, suggesting parallel local evolution of potters' skills and technical preferences, strong inter-regional interactions or both. The study of almost 500 early Neolithic pots, with a wide variety of shapes and surface treatments, analysed in thin section shows the use of almost the same fabric recipe used throughout the central Balkans (Spataro 2011a; 2011b; 2017). In the late stages of the early Neolithic, potters still used a formula that their predecessors used at the beginning of the 6th millennium BC (Spataro 2011a; 2011b). Nevertheless, they started painting their pots with Mn-rich ores, and the painted decorations were independent from the ceramic fabrics.

No correlation between fabric and function was detected in the coarse and/or painted wares (Table 5). Although the sample size is too small to draw firm conclusions, it appears that potters did not select raw materials with a specific purpose of pottery in mind.

The identification of different food sources was based on the comparison of carbon isotope composition of main fatty acids (palmitic and stearic) to those of modern reference animal fats (Fig. 10). All archaeological lipids detected derived from ruminant and non-ruminant animal fats and dairy products, in agreement with the main trend observed previously at the site (Craig et al. 2005) and at early Neolithic sites in other regions of the Balkans (Ethier et al. 2017). Most of the animal fats detected at the sites plot with the range of ruminant adipose fats (n= 6) (Fig. 10). Potential contributors of ruminant fats are both wild (aurochs, red and roe deer) and domestic (cattle, sheep and goat) animals, all of them well represented in the Neolithic archaeological levels (Bartosiewicz et al. 2006). In addition, non-ruminant adipose fats can also reflect the exploitation of wild or domestic pig (Bartosiewicz et al. 2006). Despite the wide representation of animal species in the Neolithic levels, including different fish species, there is no evidence of aquatic resources in the 16 vessels from Area VI with significant organic residues (Craig et al. 2005; this paper), in contrast to the identification of aquatic biomarkers in 5 of the 11 sherds from Area VII (Cramp et al. 2019). There is currently no evidence that pottery from these two excavation areas, 20-25m apart, represents different periods. We detected dairy products only in three polished/slipped and painted sherds (SCL4, 6, 11). This seems to reinforce the idea that polished and slipped ware were used for drinking, as the compressed surface made the vessels less permeable. Craig et al (2005) also found dairy products in a painted bowl and an undecorated amphora. Dairy products are often heated or fermented in pottery, so dairy residues should not be restricted to serving vessels. Ruminant adipose fats were detected in slipped and painted as well as plain ware (see samples SCL2 and 5). This picture is supported by use-alteration analysis of pottery from the SKC site of Blagotin in Serbia, where fine polished slipped ware was used in the same way, and therefore for similar purposes, as other pottery classes (Vuković 2011, 210).

Conclusions

For the first time, archaeometric and organic residue analyses have been applied to the same Neolithic sherds. At Schela Cladovei, plain, slipped, polished and painted SKC ceramics were made using similar pottery recipes to those that were in use throughout the SKC world, for c. 700 years (e.g. Spataro 2011; 2017; in press), with micaceous clays, chaff temper, and low firing in an oxidising atmosphere, regardless of decoration or shape. The potters used raw materials that are compatible with the local geology. The dark brown/black linear and dynamic geometric motifs, typical of the late Starčevo phases, were painted using a solution that contained high concentrations of Mn and Fe ores, mixed with clay and water. The Mn and Fe ores might have been found further north in the Carpathians and possibly locally as well. The organic residue analyses showed the cooking of dairy products and meat of domestic and/or hunted mammals, in contrast to Cramp et al's (2019) results from a nearby area of the site, where aquatic biomarkers occurred in 5 of the 11 pots with sufficient residue for analysis, and only one pot held dairy products. This contrast is currently unexplained, but our results show no correlation between biomolecular results and fabric, implying that no specific type of pot was made for a specific use, given that fabrics and shapes were not correlated in SKC pottery overall (Spataro in press). There could be a difference in date between our samples and those studied by Cramp et al. (2019), but the fragmentary condition of the sherds and lack of direct dating of the organic residues precludes discussion of temporal patterns in pottery function.

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Figure captions:

Figure 1. Aerial view of the Schela Cladovei site. The yellow boundary line delineates the area that is accessible archaeologically and where Mesolithic and/or Neolithic artefacts have been observed eroding from the

riverbank. Inset map: location of Schela Cladovei within the Starčevo-Körös-Criş culture area (hatched) (adapted from Boroneanţ & Bonsall 2013: fig. 4.1).

Figure 2. Schela Cladovei: top: samples analysed and discussed in this paper (photo by M. Spataro); bottom: other Starčevo-Criş ceramics from the site: a, rim sherd with finger/tool impressions on the rim and an appliqué band on its lower part, which is also decorated with finger impressions; b, body sherd with nail impression decoration; c, body sherd with pinched decoration; d, body sherd decorated with rows of incised parallel lines in herringbone motif; e, sherd with appliqué band decorated with 'alveoli' (finger impressions); f, body sherd with organised *barbotine* decoration; g, rim sherd with finger/tool impressions on the rim and thin incised lines crosshatched on the body (mesh or net pattern) (drawings by E. Rayner).

Figure 3. Photomicrographs of thin sections of ceramic samples from Schela Cladovei: showing (a) limestone in a non-calcareous matrix and some voids left by vegetal temper (sample SCL1); (b) fine well-sorted quartz and vegetal matter (sample SCL8); (c) vegetal matter and scattered coarser sand (sample SCL6); a slip and a painted layer are also visible (d) matrix with well-sorted and fine quartz and some clay pellets and a slip (sample SCL5). All photomicrographs were taken in cross polarised light (XPL), except photomicrograph c which was taken in plane polarised light (PPL) (photos by M. Spataro).

Figure 4. SEM BSE images of sample SCL11, showing (a) non-vitrified paste and (b) charred plant remains still present in the voids. (c) Optical microscopy image of traces of red coating still visible on sample SCL2; and (d) SEM BSE image of SCL2 showing the red coated layer on the surface, which is rich in fine iron oxides (bright spots) (photos by M. Spataro).

Figure 5. Principal Components Analysis output (Components 1 and 2), based on SEM-EDX compositional data of ceramics from Schela Cladovei. PCA was carried out using Past v. 3.18 (Hammer *et al*. 2001). Legend: (dot) Fabric group 1; (triangle) Fabric group 2; (square) Fabric group 3. Each point represents the average of four bulk analyses.

Figure 6. Sample SCL5: an image of its surface (left) and (right) optical microscopy image of the painted surface, slip and body paste (photos by M. Spataro).

Figure 7. Sample SCL5: optical microscopy image of painted surface (a) and (b) of the thin section (with the painted are, dark coating at the top, the slip (red interlayer) and the body paste); (c) SEM-EDX elemental x-ray map, mapped area of sample SCL5 (x150), including painted layer (bright area) and slipped area (between the paint and the body paste); (d) elemental map of iron; e) elemental map of manganese; f) elemental map of carbon. Colours representing abundance, from absent (black) to very abundant (white) (for colour scale, see legend) (photos by M. Spataro).

Figure 8. SEM-EDX elemental x-ray map of sample SCL6 in polished thin section: a. BSE image of the mapped area (black paint [bright area] and body paste); concentrations of six elements (b. aluminium, c. silicon, d. iron, e. potassium, f. magnesium, and g. calcium), colours representing abundance, from absent (black) to very abundant (white) (for colour scale, see legend); h. polarised light micrograph, showing the mapped area. (Photos by M. Spataro).

Figure 9. SEM BSE images of (a) sample SCL6, showing the body paste and the painted layer (bright area in the centre) and some post-depositional material on the painted layer and (b) sample SCL9, showing the black painted layer at high magnification (photos by M. Spataro), Scales in microns.

Figure 10. $\delta^{13}C$ values of $C_{16:0}$ and $C_{18:0}$ n-alkanoic acids extracted from pottery sherds from Schela Cladovei (Area VI: Craig et al., 2005; this study; Area VII: Cramp et al., 2019). The 68% confidence ellipses are based on modern reference fats (ruminant, non-ruminant, dairy and marine resources) from the European continent (Dudd, 1999; Spangenberg et al., 2006, 2010; Craig et al., 2011, 2012; Spiteri, 2012; Cramp et al., 2014; Carrer et al., 2016) corrected for the Suess Effect taking into consideration the date of collection (Hellevang and Aagard, 2015).

Table captions:

Table 1. Schela Cladovei: list of fabric groups, clays and temper types, surface treatment, analyses carried out and year of excavation and sherd label.

Table 2. Compositional data from SEM-EDX analysis of the fabrics of Schela Cladovei samples (grey rows, mean of four bulk analyses) and standard deviation (white rows). Results are reported as normalised oxides; - indicates below detection limit.

Table 3. Compositional data from SEM-EDX analysis of the pigments identified on the surfaces of the Schela Cladovei samples. Shaded rows: slips/red coatings; white rows: paint. Results are reported as normalised oxides; - indicates below detection limit.

Table 4. Molecular and isotopic information from pottery sherds from Schela Cladovei.

Table 5. Fabric vs main function of the samples analysed in this study.