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Postal address (materials sent for reviewing purposes and other correspondence):
Universitatea “Al. I. Cuza”, Facultatea de Istorie, Bulevardul Carol I, nr. 11, 700506 – Iași, Romania.
Tel.: (+04) 0232 201 615; Fax.: +(4) 0232 201 201, +(4) 0232 201 156;
Website: saa.uaic.ro; Email: saa.uaic.ro@gmail.com, blucretiu@yahoo.com.

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Same or different? Interdisciplinary analyses on the Costișa and Monteoru pottery from Siliștea-*Pe Cetățuie* settlement

Ana DROB[1], Neculai BOLOHAN[2], Bogdan RĂȚOI[3], Sebastian DROB[4]

Abstract: *The archaeometric study of prehistoric pottery is an effective tool for investigating this abundant category of artifacts from an archaeological site. In this study, the physicochemical analyses used are optical microscopy (OM), scanning electron microscopy (SEM), coupled with energy dispersive X-ray analysis (EDX) and Micro-Fourier Transform Infrared Spectroscopy (μFTIR). Through these techniques were studied 48 ceramic fragments belonging to the Costișa and Monteoru ceramic groups from the Middle Bronze Age settlement from Siliștea-Pe Cetățuie. Also, the ceramic fragments were investigated macroscopically, using criteria such as color, production technique, type, size, functionality and category of the vessel. The obtained results provided important data related to the ceramic technologies of the two communities from the studied settlement.*

Rezumat: *Studiul arheometric al ceramicii preistorice este un instrument eficient pentru investigarea acestei abundente categorii de artefacte dintr-un sit arheologic. În acest studiu, analizele fizico-chimice utilizate sunt Microscopia Optică (OM), Microscopia Electronică de Scanare (SEM) cuplată cu analiza de raze X cu Dispersie de Energie (EDX) și Spectroscopia în Infraroșu cu Transformata Fourier (μFTIR). Prin aceste tehnici au fost studiate 48 de fragmente ceramice aparținând grupurilor ceramice Costișa și Monteoru din așezarea de bronz mijlociu de la Siliștea-Pe Cetățuie. De asemenea, fragmentele ceramice au fost investigate macroscopic, folosind criterii precum culoarea, tehnica de realizare, tipul, dimensiunea, funcționalitatea și categoria vasului. Rezultatele obținute au furnizat date importante legate de tehnologiile ceramice ale celor două comunități din așezarea studiată.*

Keywords: pottery, archaeometric analysis, Middle Bronze Age, OM, SEM-EDX, μFTIR

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[1] Institute of Interdisciplinary Research, Department of Exact Sciences and Natural Scince, Arheoinvest Center, ”Alexandru Ioan Cuza” University of Iași; anadrob1@gmail.com
[2] Faculty of History, ”Alexandru Ioan Cuza” University of Iași; neculaibolohan@yahoo.com
[3] Faculty of Geography and Geology, Department of Geology, ”Alexandru Ioan Cuza” University of Iași
[4] SC Banat Archaeosave SRL

Introduction

Siliștea-*Pe Cetățuie* site is located in the south-eastern extremity of the Cracău and Bistrița rivers watersheds (Map 1), in the southern part of the former village of Bârjoveni, today unified with Siliștea (Români administrative unit, Neamț county–GPS: N 46°47´43.81"; E 26°43´33.00"). The settlement is located on a high area, with good visibility, situated near the confluence of the Bistrița and Siret rivers, both hydrographic arteries being important communication routes in the eastern Carpathian area. In the fortified Middle Bronze Age settlement from Siliștea was identified a single level of habitation in which Costișa and Monteoru Ic3-Ic2 archaeological materials were discovered, without a stratigraphical difference (1955/1739-1614/1773 BC)[5].

The *Cetățuia* plateau from Siliștea has an altitude of 448.66 m and an NNW-SSE orientation. The settlement is naturally defended from three directions (N, E, W) by steep slopes, with an inclination of 25-37°, which creates accessibility difficulties[6]. The defensive system was completed by an anthropic ditch, with a depth of 3.20 m and an opening of 15 m, that was covered with sandstone from the geological structure of the hill. Following the non-invasive research carried out in the Siliștea-*Pe Cetățuie* site were identified two more defensive structures, such as ditches, which are located inside the settlement and were covered from antiquity, leaving no elevation marks[7].

During the archaeological researches from the Siliștea-*Pe Cetățuie* settlement, were discovered several rectangular, unburned housing structures, represented by large sandstones that formed the basis of support pillars, as well as red-burned sandstones, adobe and ceramic fragments. Also, there were identified and some elements that indicate the practice of a foundation ritual of these constructions, being discovered four such situations in which, under the large sandstones, were discovered whole or broken vessels, as well as animal bones that represented meat offerings[8].

Regarding the archaeological materiality of this settlement, in addition to the multitude of bone (pointed bone tools, pendants, arrowheads), stone (axes, curved knives, arrowheads, grinders) and clay artifacts (spindle whorls)[9], were discovered several bronze objects represented by six *Noppenringe*, a simple ring, bracelet and a small circular ring[10]. The metal objects have analogies in Central Europe, illustrating the existence of contacts or relations of the communities from Eastern Carpathians with the Middle Danube area[11].

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5 BOLOHAN, 2010, 238-239.

6 BOLOHAN, GAFINCU, 2018, manuscript; BOLOHAN *et al.*, 2019, manuscript.

7 BOLOHAN, 2016, 75-78.

8 BOLOHAN, MUNTEANU, 2001, 46; BOLOHAN, CREȚU, 2004, 57-58; BOLOHAN, GAFINCU, 2018, manuscript.

9 BOLOHAN, GAFINCU, 2017, manuscript; BOLOHAN, GAFINCU, 2018, manuscript; BOLOHAN *et al.*, 2019, manuscript.

10 BOLOHAN *et al.*, 2019, manuscript.

11 BOLOHAN, 2003, 197; BOLOHAN, CREȚU, 2004, 59-60.

The special relations that the two communities from the Siliștea-*Pe Cetățuie* site had, constitutes a point of particular interest of this approach. If in some settlements the stratigraphic report is not known, and in others it is unclear, the housing situation from Siliștea can be a clarifying element in this regard. Thus, the study of the pottery of the two ceramic groups by analytical methods can provide important information on the nature of the relations between these communities by means of technological knowledge involved in pottery manufacture. So far, interdisciplinary analyses have been performed before for the ceramics of these two communities from several settlements in the Bistrița River basin, which represent a database for Middle Bronze Age pottery in this microzone[12].

Methodology

In this paper will be analysed 24 Monteoru ceramic fragments Monteoru (1-24M) and 24 Costișa pottery fragments (1-24C), marked **Slș**. (Siliștea), for which will be performed macro- and microscopic observations, as well as scanning electron microscopy (SEM) coupled with energy-dispersive X-ray analysis (EDX) and micro-Fourier transform infrared spectroscopy (μFTIR)[13]. Also, the statistical representation of the functional classes was transposed in the selection of the samples for analysis. Thus, we tried to keep the proportions, but on a small scale, selecting a representative number of ceramic fragments from each functional category. The macroscopic analysis[14] was performed for each functional category of vessels identified in the settlement, both for Costișa and Monteoru pottery, considering information such as color, wall thickness, hardness, surfaces and applied treatments, firing type, kneading and inclusions (type, size, shape, frequency, sorting).

The microscopic analysis of the fragments was performed with a Zeiss Imager.a1M microscope with a built-in AXIOCAM camera, which uses AxionVisionRelease 4.7.1 software. The samples were sanded with a Stroers LaboPol device using discs with different granulations.

In the current analyses was used an electron microscope with SEM scan, model VEGA II LSH, produced in the Czech Republic by TESCAN, coupled with an EDX detector type QUANTAX QX2, produced in Germany by BRUKER/ROENTEC. The SEM micrographs consisted of backscattered electrons (BSE) at 200× magnifications for ceramic paste and surfaces, and the samples were not covered with metal or graphite.

The spectra were recorded with an FTIR spectrophotometer coupled with a HYPERION 1000 microscope, both equipment from Bruker Optic, Germany. The FTIR spectrophotometer is of

12 BOLOHAN, 2013a, 199-239; BOLOHAN, 2013b, 33-56; DROB, 2019, 197-214; DROB, 2021, 251-275; DROB *et al.*, 4885, 1-17; DROB, 2021.

13 The analyses presented in this paper, as well as the illustration, are found in another, more extensive form in DROB, 2021.

14 SHEPARD, 1954; RYE, 1981; RICE, 1987; ORTON *et al.*, 1993; GOFFER, 2007; PREHISTORIC CERAMICS RESEARCH GROUP, 2010.

the TENSOR 27 type, which is predominantly suitable for measurements in close IR. The standard detector is DLaTGS covers the spectral range 4000–600 cm^{-1}, and works at room temperature. The resolution is usually 4 cm^{-1} but can also reach 1 cm^{-1}. The detector is of the MCT type cooled with liquid nitrogen (-196°C) and the measured area is optimized to a diameter of 250 μm, reaching a minimum of 20 μm.

Results and Discussions

A. Macroscopic analysis

The studied Costișa ceramic fragments come from four cooking vessels, from seven food preparation, serving and consumption vessels, from six solid and liquid storage vessels and from five storage and transportation liquids vessels. Also, one ceramic fragment was selected from a special purpose pot and one from a multi-purpose vessel.

Regarding the colors of the ceramic fragments, in the case of Costișa pottery, the outer surfaces are mostly brown, light brown and reddish yellow, but there are also colors such as intense brown, yellowish brown, gray, dark gray and very dark gray. Inner surfaces, generally, have the same colors but with fewer shades, and in this case, brown is the main color, followed by reddish yellow, light yellowish brown and very dark gray, but also some fragments that have dark gray and yellowish-brown interiors (Munsell Soil-Color Charts). The vessels color variety indicates an uncontrolled firing, which did not allow to obtain unitary chromatic pots.

Pots from the cooking functional category (Slș.C1–Pl.1/1; Slș.C2–Pl.1/2; Slș.C3–Pl.1/3; Slș.C4–Pl.1/4) were burned in a reducing (Slș.C1, Slș.C3, Slș.C4) and incomplete oxidizing (Slș.C2) atmosphere, resulting in hard vessels, with a semi-fine surface and a very well finished exterior, with no treatment applied, only two pots (Slș.C1, Slș.C4) having incised decoration at the top. Also, the interior surfaces are well smoothed and finished. The wall thickness is between 7.35-12.59 mm, and the macroscopically visible inclusions are the ceramoclasts, of medium size and a distribution of 10-15%, all having a rounded shape and being well sorted. In the case of the Slș.C2 fragment, three large lithoclasts are also visible, these being considered accidental, and are also visible large cracks on the inner surface produced by heat exposure. Another proof in this sense is represented by the smudging traces in area between upper volumes (Fig.1).

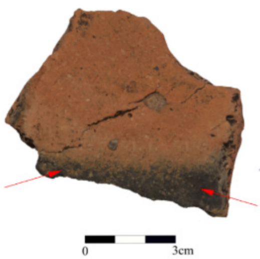
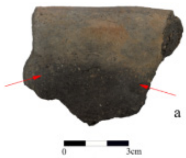


Fig. 1. Slș.C2 fragment with cracks caused by heat exposure and smudging traces (Own archive image)

The functional class of food preparation, serving and consumption is represented by seven vessels (Slș.C8–Pl.1/5; Slș.C11–Pl.1/6; Slș.C5–Pl.1/7; Slș.C9–Pl.1/8; Slș.C10–Pl.1/9; Slș.C6–Pl.1/10; Slș.C7–Pl.1/11) which were fired in a reducing environment, with two exceptions, fired in an oxidizing atmosphere (Slș.C5, Slș.C6). The exterior surfaces are well smoothed (Slș.C5, Slș.C6-C9), being identified the application of a ceramic slip (Slș.C10, Slș.C11). The inner surfaces are well and very well finished, some with fine cracks caused by inclusions. In the case of the Slș.C6 fragment, strong smudging traces are visible on the inside (Fig.2/a), and the Slș.C8 vessel exterior shows secondary firing spots that have penetrated the vessel wall, being observable also on the inner surface (Fig.2/b). These traces indicate the use of these pots for cooking, suggesting food preparation with fire. One of the vessels was decorated entirely (Slș.C10) and four other have only auxiliary elements (handles) in the opening area. The fragments are hard, with a fine (Slș.C8, Slș.C9, Slș.C11) and semi-fine surface. In the paste, but also on the surfaces, are visible the ceramoclasts and in four samples the carbonates (Slș.C7, Slș.C9-C11). The wall thickness of these vessels is between 7.45-13.20 mm, the inclusions having medium size, sub-rounded shape and a frequency of 15-20%, being well integrated in the clay mass.

The category of vessels for storage of solid or liquid goods includes six containers (Slș.C15–Pl.2/2; Slș.C13–Pl.2/1; Slș.C14–Pl.2/5; Slș.C16–Pl.2/3; Slș.C17–Pl.2/4; Slș.C12–Pl.2/6). They were fired in an oxidizing (Slș.C12, Slș.C13, Slș.C17), reducing (Slș.C14, Slș.C16) and mixed (Slș.C15) atmosphere, resulting in hard vessels with semi-fine and semi-coarse (Slș.C12, Slș.C15) surfaces. Some pots have a carelessly finished (Slș.C12) or only smoothed (Slș.C13, Slș.C15, Slș.C16) exterior, being identified also well-finished and very well-finished specimens (Slș.C14, Slș.C17). The applied decoration is found on a single vessel (Slș.C15), with its finishing traces, and the auxiliary elements (handles) are found on two other containers (Slș.C13, Slș.C14). The inner surfaces are well finished and in most specimens are visible fine cracks produced by inclusions. The wall thickness is between 7.93-13.09 mm and in the paste of the vessels are visible ceramoclasts and carbonates (Slș.C13, Slș.C16). The inclusions have medium dimensions, sub-rounded and sub-angular shape and a distribution of 15-20%, being, in general, well integrated in the clay matrix.

The functional class for storage and transportation of liquids is represented by five vessels (Slș.C19–Pl.2/7; Slș.C24–Pl.2/9; Slș.C23–Pl.2/8; Slș.C18–Pl.2/10; Slș.C20–Pl.2/11), these being fired in a reducing (Slș.C20-C22, Slș.C24), mixed (Slș.C18, Slș.C23) and incomplete oxidizing (Slș.C19) atmosphere. The fragments are hard with a semi-fine surface (Slș.C18-C20, Slș.C22) and very hard with a fine surface (Slș.C23, Slș.C24), and have a very well finished exterior or an exterior ceramic slip (Slș.C20, Slș.C24). The inner surfaces are well smoothed and rarely well finished. The wall thickness is between 7.80-13.02 mm and the main inclusions are medium size ceramoclasts, sub-rounded shape with a frequency of 10-15%.



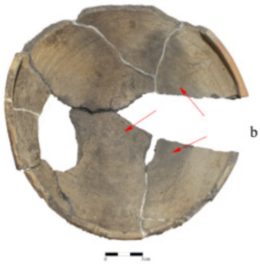


Fig. 2. Smudging traces (a-Slș.C6) and secondary firing spots (b-Slș.C8) identified on the preparation, serving and consumption vessels from Siliștea-*Pe Cetățuie* settlement (Own archive images)

The special purpose pot (Slș.C22–Pl.2/12) represents a miniature of the storage and transportation liquids vessels, being fired in a reducing environment, resulting in a hard vessel. The outer surface was covered with a ceramic slip and the interior was well finished. In the upper part, in the opening area, the vessel still keeps the trace of a handle that was broken from antiquity, this being doubled, most probably, by the existence of a second handle. The wall thickness is 5.10 mm and in the paste were identified small ceramoclasts, with rounded shape and a low frequency of 5-10%. The clay matrix and the constituent elements were well kneaded and homogenized, being visible only the secondary pores.

The multi-purpose vessel (Slș.C21–Pl.2/13) was fired in a reducing atmosphere, is very hard and has a semi-fine surface, being very well finished. The pot was decorated in the opening area with incised motifs, these being very carefully made. The wall thickness is 4.20 mm, and the paste from which it was manufactured contains small, rounded ceramoclasts, with a distribution of 5-10%. These inclusions are very well integrated into the clay mass, indicating very good kneading.

The Monteoru fragments come from two cooking vessels, from five food preparation, serving and consumption vessels, from eight solid or liquid goods storage containers, from six storage and transportation liquid vessels and from three multi-purpose pots.

The colors of the Monteoru analysed vessels indicate a greater variety of shades than in the case of Costișa ones. Thus, the main exterior color is reddish yellow, followed by brown, strong brown, light reddish brown, reddish brown, gray, pinkish gray, dark reddish gray, dark gray and very dark gray. The inner colors are characterized by a greater variety of shades, but most of them are brown, followed by strong brown, reddish yellow, light brown, reddish brown, light reddish brown, light yellowish brown, gray, dark reddish gray, reddish gray, pinkish gray, very dark gray and a fragment with a yellowish red interior (Munsell Soil-Color Charts). Therefore, as in the case of Costișa, Monteoru pottery also shows chromatic clues that suggest an uncontrolled firing of the vessels.

The category of cooking vessels is represented by two pots (Slș.M1–Pl.3/1; Slș.M2–Pl.3/2), which were fired in a reducing atmosphere followed by an oxidizing one (Slș.M2) and in a mixed environment (Slș.M1), being hard, with semi-fine and very well finished surfaces. Also, the interior of the vessels is well finished, being observed fine cracks on one of the containers (Slș.M1). As regards the decorative elements, only one container shows embossed motifs in the supramedian area. The wall thickness is between 5.90-6.59 mm, being visible medium-sized ceramoclasts, sub-rounded shape, with a distribution of 10-15%. They are well integrated in the clay matrix, which indicates a good kneading, resulting only secondary pores.

The functional category of food preparation, serving and consumption is represented by five vessels (Slș.M6–Pl.3/6; Slș.M7–Pl.3/7; Slș.M3–Pl.3/3; Slș.M4–Pl.3/4; Slș.M5–Pl.3/5), these being fired in a reducing (Slș.M3, Slș.M4, Slș.M7), mixed (Slș.M5) or incomplete oxidizing (Slș.M6) atmosphere. The fragments are hard, with a semi-fine and fine surface, being well finished (Slș.M4, Slș.M5), very well finished (Slș.M3, Slș.M6) or having applied an exterior ceramic slip (Slș.M7). The interior of the vessels is well smoothed and finished, being identified two pots (Slș.M3 and Slș.M5) with secondary firing spots that penetrated the vessel's wall (Fig.3/a and b), indicating the use of these containers in food preparation with a source of heat. Two of the vessels have a decoration applied in the upper part, being visible also the traces of smoothing and finishing of these elements. The wall thickness varies between 7.20-10.02 mm and the inclusions identified in the ceramic paste are the ceramoclasts, of medium size, with sub-rounded shape and a frequency of 10-15%.

The category of vessels intended for storage of solid or liquid goods is represented by eight pots (Slș.M12–Pl.3/8; Slș.M8–Pl.3/10; Slș.M10–Pl.3/9; Slș.M13–Pl.3/11; Slș.M14–Pl.3/12; Slș.M15–Pl.3/13; Slș.M9–Pl.3/15; Slș.M11–Pl.3/14) fired in an oxidizing (Slș.M9, Slș.M10, Slș.M12-Slș.M14) and incomplete oxidizing (Slș.M8, Slș.M11, Slș.M15) atmosphere, resulting in hard containers. The surfaces of the vessels are semi-fine and very well finished (Slș.M9-Slș.M11, Slș.M15) or with external ceramic slip (Slș.M8) and semi-coarse that are only well smoothed (Slș.M12, Slș.M13, Slș.M14). Most of the pots have a decoration applied in the supramedian area, one of them also having auxiliary elements such as handles (Slș.M10), and two other vessels are undecorated (Slș.M9, Slș.M11). The wall thickness is between 9.37-12.73 mm and in all fragments are visible ceramoclasts, with medium and large dimensions, sub-rounded and sub-angular shape, with a distribution of 15-20%. All identified inclusions are well sorted and homogenized in the clay paste.

From the category of vessels intended for storage and transportation of liquids were selected six fragments (Slș.M16–Pl.4/1; Slș.M19–Pl.4/3; Slș.M20–Pl.4/4; Slș.M17–Pl.4/2; Slș.M18–Pl.4/6; Slș.M21–Pl.4/5). Of these, four pots were fired in a reducing environment (Slș.M16, Slș.M18-M20), one was fired oxidizing (Slș.M17) and one in an incomplete oxidizing

(Slș.M21) atmosphere. Some of the containers are hard with a fine (Slș.M17, Slș.M21) and semi-fine (Slș.M19) surface and some are very hard with a semi-fine surface (Slș.M16, Slș.M18, Slș.M20).

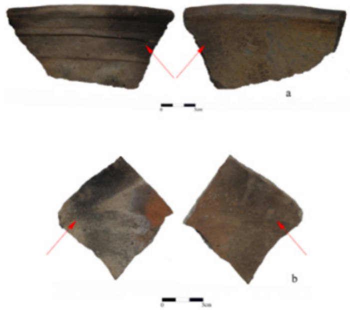


Fig. 3. Monteoru vessels from the preparation, serving and consumption functional class with secondary firing spots from Siliștea-Pe *Cetățuie* settlement: a-Slș.M3; b-Slș.M5 (Own archive images)

The outer surfaces are very well finished or have applied a ceramic slip (Slș.M18, Slș.M21), while the interior is finished more carelessly, but well smoothed. The vessels of this functional category are also decorated by incision (Slș.M16, Slș.M17), but most of them have applied decoration in the area between the upper volumes (Slș.M18-M21), as well as auxiliary elements (handles) in the submedian area (Slș.M18, Slș.M21). The wall thickness is between 6.00-7.81 mm, and the used inclusions are medium-sized ceramoclasts, sub-rounded shape and a distribution of 10-15%, being well integrated in the clay mass.

The category of multi-purpose vessels is represented by three containers (Slș.M22–Pl.4/9; Slș.M23–Pl.4/7; Slș.M24–Pl.4/8), these being fired in an oxidizing (Slș.M22) and reducing (Slș.M23, Slș.M24) environment, resulting in hard vessels. The surfaces of the fragments are semi-fine and very well finished (Slș.M22, Slș.M23) and fine having applied an exterior ceramic slip (Slș.M24), the interior being well smoothed and finished. Two of the containers were decorated in the supramedian area, one with incision (Slș.M23) and the other with embossed motifs (Slș.M22). The wall thickness is between 5.15-7.70 mm, being visible the medium-sized, sub-rounded ceramoclasts, with a frequency of 10-15%. Kneading and homogenizing the paste is very good, resulting only secondary pores.

The macroscopic analysis performed for the two ceramic groups from the Siliștea-*Pe Cetățuie* settlement highlighted a series of common aspects related to the pottery manufacturing in this site. Thus, it was noted that all the vessels were made by the coiling technique, identified by means of the fine bumps on the inner surfaces or by the joining marks on the edges of the ceramic fragments. The auxiliary elements (handles) that appear on some pots were made by modeling and, most of the time, are visible the finishing traces.

The outer and inner surfaces of the vessels are generally very well finished, with no traces of burnish. In some cases, the ceramic slip appears as an exterior treatment, which has been identified in all the functional categories of both pottery ensembles. Also, on the cooking pots and on the preparation, serving and consumption vessels are visible smudging traces on the inside, as well as secondary firing spots on the outside, which penetrated the containers wall, indicating the use of these pots in hot food preparation.

The colors and shades of the vessels are mostly different, but they seem to be more unitary in the case of Costișa. The chromatic variety of the containers may indicate a poorly controlled firing which does not allow homogeneous colors. The fact that in no functional class of pottery, both Costișa and Monteoru, seems to be no connection between the type of vessel and color, can indicate a firing of the pots together, without preferring a special treatment for certain functional categories.

In all the studied fragments the ceramoclasts are present as intentional inclusions, and thus were identified three types of paste for both communities pottery, differentiated according to their quantity, shape and distribution. Regarding Costișa pottery, the first category, marked *SCCP1* (Siliștea-Costișa Paste Category 1) defines a paste with small ceramoclasts, rounded shape and with a reduced distribution of 5-10%, this being used in the manufacturing of special and multi-purpose vessels. The second category, *SCCP2*, includes medium-sized, sub-rounded and sub-angular ceramoclasts, which have a frequency of 10-15%, being found in the paste of cooking vessels and in those for transporting and storing liquids. The third category, *SCCP3*, is characterized by medium and large ceramoclasts, sub-rounded and sub-angular, with a distribution of 15-20%, being characteristic for the preparation, serving and consumption vessels and for the storage of solid and liquid goods pots. In the case of Monteoru pottery, the first category, marked *SMCP1* (Siliștea Monteoru Pasta Category 1) includes small, rounded ceramoclasts, which have a distribution of 5-10%, being characteristic for the multi-purpose vessels. The second category, *SMCP2*, includes medium-sized, sub-rounded and sub-angular ceramoclasts, with a frequency of 10-15%, being used to make cooking, preparation, serving and consumption vessels and for those of transportation and storage of liquids. The third category of paste, *SMCP3*, is characterized by medium and large ceramoclasts, sub-rounded and sub-angular, with a frequency of 15-20%, specific to storage vessels.

B. Interdisciplinary analyses

B.1. Microscopical and mineralogical analysis

Optical microscopy performed on Costișa samples (Fig.4/left) showed the presence of natural minerals from the used clay, such as quartz and mica, but also of intentional inclusions, such as ceramoclasts. The reused ceramoclasts were also identified in 13 fragments (Slș.C1-C6, Slș.C9, Slș.C12, Slș.C13, Slș.C15, Slș.C17-C19). The carbonates are visible in five samples (Slș.C9, Slș.C11, Slș.C13, Slș.C16, Slș.C18) suggesting a low firing temperature that did not allow their decomposition. Another indication of relatively low firing temperatures is represented by the traces of organic matter identified in six samples, visible in the form of pores or black lamellae (Slș.C13-C15, Slș.C17, Slș.C20, Slș.C23). Also, other natural inclusions observed in the ceramic paste are represented by iron oxides, that are visible in all fragments, having appreciable dimensions or being discrete presences.

The Monteoru fragments analysed by optical microscopy (Fig.4/right) contain, as in the case of Costișa, minerals specific to the raw material, such as quartz, mica and iron oxides, which in some samples have great dimensions. The ceramoclasts are also present in the paste of Monteoru pottery, being also identified 14 fragments that contain reused ceramoclasts (Slș.M1-M3, Slș.M5, Slș.M8-M12, Slș.M14, Slș.M16, Slș.M21, Slș.M23). The organic matter is observable in six samples (Slș.M2, Slș.M6, Slș.M9, Slș.M10, Slș.M14, Slș.M22), and the carbonates in seven fragments (Slș.M5-M7, Slș.M13, Slș.M16, Slș.M19), which suggests relatively low firing temperatures.

For mineralogical information were made four thin sections, two for Costișa pottery (Slș.C9 - Fig.5/left; Slș.C23 - Fig.5/right) and two for Monteoru pottery (Slș.M2 – Fig.6/left; Slș.M15 – Fig.6/right). From a geological point of view, on the plateau of the settlement are Quaternary deposits belonging to the Middle Pleistocene, represented by sands, gravels, boulders and loessoid deposits. Near the settlement are found Neogene Tortonian sedimentary deposits made up of sands, marly clays, gypsum and tuffs[15].

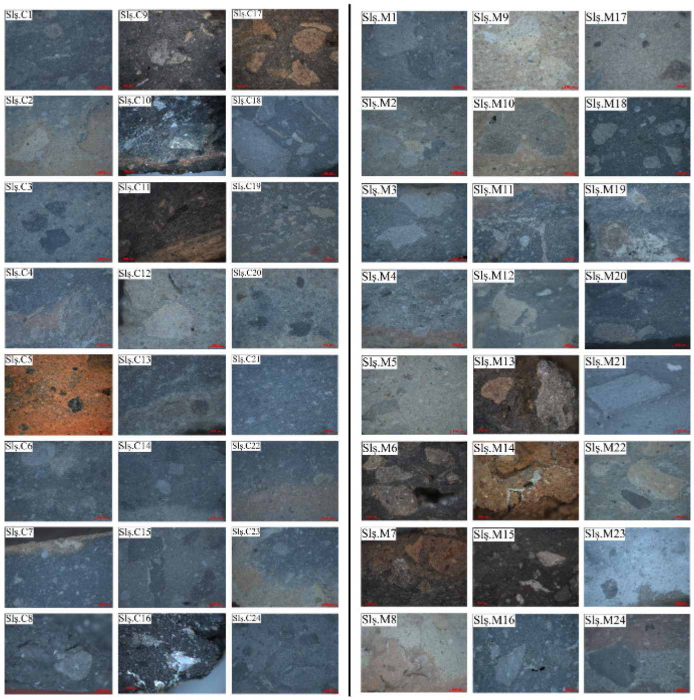


Fig. 4. Optical microscopy for the Costișa (left) and Monteoru (right) ceramic fragments from Siliștea-*Pe Cetățuie* settlement (50× magnifications)

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15 Geological Map of Romania, Scale 1:200000.

For all the samples, was highlighted the presence of subangular monocrystalline quartz and polycrystalline quartz crystals, resulted from pottery firing. In addition, muscovite, plagioclase feldspars, and hematite aggregates were identified. Moreover, in both samples the presence of ceramoclasts and main pores resulting from clay kneading is noticeable. In addition, a limestone lithoclast (Sl.C9) and a sandstone (Sl.C23) were also identified, all these minerals corresponding to the geological features of the studied fragments area of provenance.

Following the mineralogical analysis performed for the four pottery fragments, were obtained some information regarding the clay used in the pottery manufacture. Thus, in this process was used a local quartz clay with amorphous appearance and semi-oriented texture, indicating a semi-fine to fine ceramic, in which were added intentional inclusions of ceramoclast type.

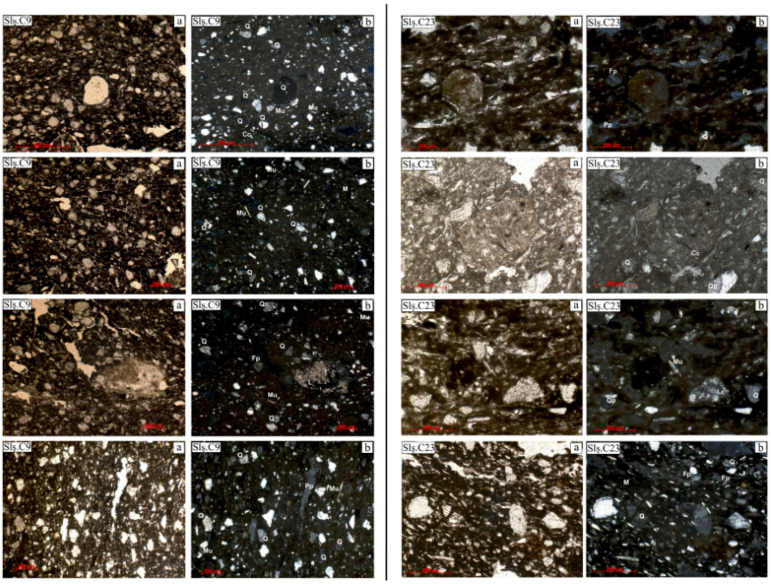

Fig. 5. Mineralogical section of the Slș.C9 fragment (left): Q-quartz; Mu-Muscovite; Cc-Ceramoclast; Fp-Feldspar plagioclase; L-Limestone lithoclast; Pp-primary pores (a-parallel nicols; b-crossed nicols); Mineralogical section of the Slș.C23 fragment (right): Q-quartz; Qp-polycrystalline quartz; Mu-Muscovite; Cc-Ceramoclast; Fp-feldspar plagioclase; Lc-sandstone lithoclast; Pp-primary pores (a-parallel nicols; b-crossed nicols)

Microscopic analysis performed for all ceramic fragments showed the presence of minerals specific to the raw material, such as quartz, mica and iron oxides. These results were also confirmed by mineralogical observations, which also highlighted the presence of feldspars and indicated the use of a local quartz clay. By optical microscopy were identified the reused ceramoclasts, both in the paste of Costișa and Monteoru vessels. This may indicate an interest about the integration of broken ceramics in the paste of the new pots, action that could represent a "recycling mechanism", which also has important technological effects on pottery, as well as a specific role, representing a way of perpetuating cultural identities and ideas. The presence of carbonates and organic matter, which may or may not be intentional, is an important clue of the obtained firing temperatures, which did not exceed 700-750°C, when the carbonate type compounds are completely removed from the ceramic paste.

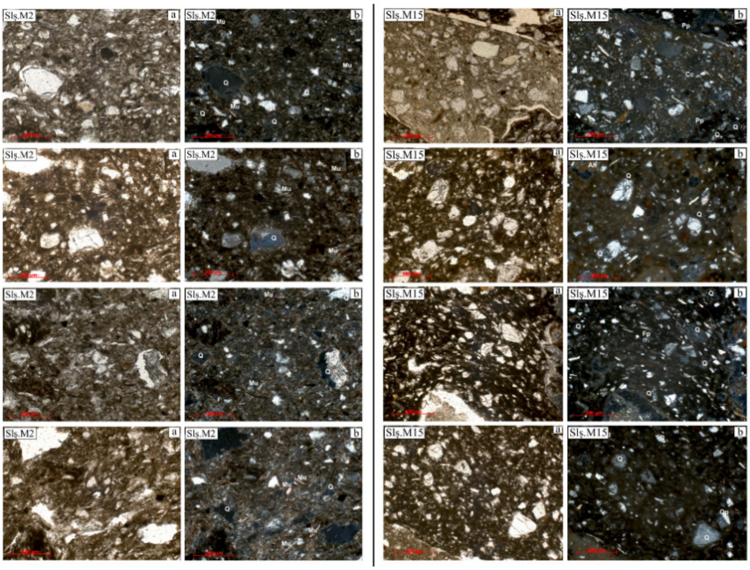

Fig. 6. Mineralogical section of the Slș.M2 fragment (left): Q-quartz; Mu-Muscovite (a-parallel nicols; b-crossed nicols); Mineralogical section of the Slș.M15 fragment (right): Q-quartz; Qp-polycrystalline quartz; Mu-Muscovite; Ah-hematite aggregates; Cc-ceramoclast; Fp-feldspar plagioclase; Pp-primary pores (a-parallel nicols; b-crossed nicols)

B.2. SEM-EDX Analysis

The SEM results obtained for the Costișa sample surfaces showed the presence of three types of microstructural finishes (Fig.7). In the first type were identified a series of very well finished but not burnished fragments, that have closed pores and mineral granules fully integrated into the clay mass, having a compact and smooth appearance (Slș.C1I+E, Slș.C3I, Slș.C5I+E, Slș.C6E, Slș.C7I, Slș.C8I, Slș.C9E, Slș.C11I, Slș.C12E, Slș.C14I+E, Slș.C16I, Slș.C17I+E, Slș.C21E+I, Slș.C22E, Slș.C23E), belonging to all functional categories. The second type is less well finished, with relatively smooth surfaces, but where the microgranules are partially integrated in the clay mass, being visible uneven surfaces. Also, this type of finishing also has very fine cracks or larger pores resulting from the dislocation of minerals during smoothing. This category includes most of the fragments analyzed except for the very fine or coarse ones. Thus, the coarse finish is visible only in three samples (Slș.C2I+E, Slș.C4I+E, Slș.C10I+E), where the surfaces are irregular and the microgranules very well individualized. Also, on the outer surface of three pots (Slș.C1, Slș.C5, Slș.C17) are visible small black spots associated with carbon deposits resulted during vessel use near a heat source[16].

[16] ŁACIAK *et al.*, 2019, 427, fig. 4/g-l, 473.

SEM micrographs for the paste of Costișa fragments showed a good homogeneity, with microstructural elements incorporated in the clay matrix (Fig.7). In several samples (Slș.C7, Slș.C9-Slș.C11, Slș.C13, Slș.C16, Slș.C18) the homogeneity is lower, being present large pores and microgranules that are very well individualized[17]. In other two samples (Slș.C15, Slș.C17) are visible traces of burnt vegetal remains. Also, in some samples are visible compact lamellar structures, with elongated pores that point out the use of the coiling technique[18] in vessels manufacturing (Slș.C4, Slș.C7, Slș.C13, Slș.C17, Slș.C22). However, the vitrification process[19] was not identified in any of the samples, indicating that the firing temperatures did not exceed 800-850°C.

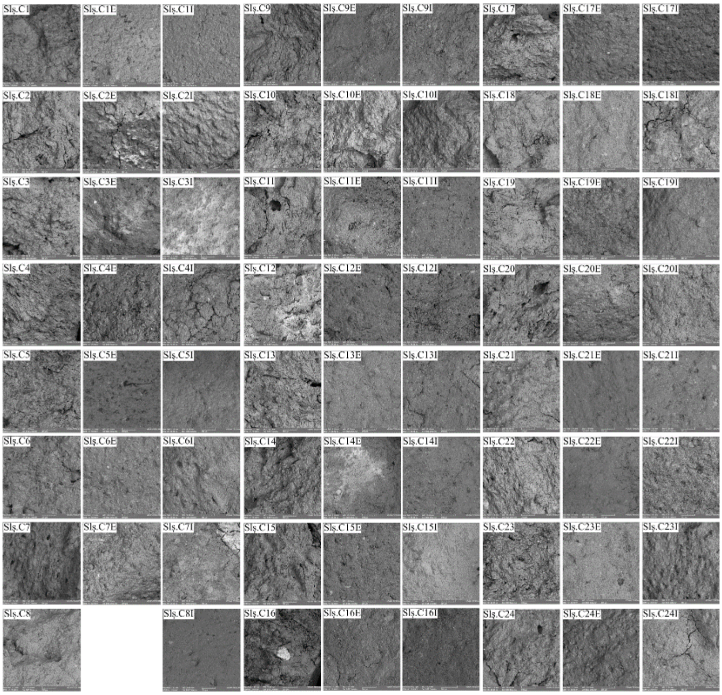

Fig. 7. SEM micrographs of the Costișa ceramic fragments (Slș.C1-Slș.C24) from Siliștea-*Pe Cetățuie* settlement (200× BSE)

Chemical composition of the Costișa paste samples (Table 1) contains elements specific to the raw material, such as silicon, aluminum, phosphorus, magnesium, calcium, potassium, sodium, iron, titanium, oxygen or carbon, attributed to aluminosilicates, quartz, oxides iron, feldspars and other mineral components present in clay. The elements that have archaeometric value and provide a series of data about the nature of the clay used, the firing temperature and the functionality of the vessels are iron, calcium, phosphorus and carbon. In

17 MANIATIS *et al.*, 1983, 777-778; PALANIVEL, MEYVEL, 2010, 340.

18 PALANIVEL, MEYVEL, 2010, 340.

19 MANIATIS, TITE, 1981, 61; MANIATIS *et al.*, 1983, 777-778; KARAPUKAITYTĖ *et al.* 2006, 386-387, Fig. 7-8; VELRAJ *et al.*, 2009, 731; AMICONE *et al.*, 2020, 14, Fig. 6/f-g.

connection with the compositional analysis of the ceramic fragments, were investigated four soil samples from the settlement, two (P1, P2) from the research unit Cas.C 2017-2019 and two (P3, P4) from the research unit SIII/2017-2018, being used as control samples, representing the basis for comparison and interpretation of the results obtained following the EDX analysis.

Table 1. The elemental composition of the Costișa ceramic paste from Siliștea-*Pe Cetățuie* settlement

	Elemental Composition in Weight Percent (%)											
Costișa Samples	Si	Al	Mg	P	Ca	K	Na	Fe	Ti	O	C	Total
Slș.C 1	23.55	10.28	1.34	2.24	2.51	2.41	0.59	5.00	0.67	51.35	0	100
Slș.C 2	19.29	10.25	1.49	5.22	4.01	1.76	0.55	4.95	0.66	51.82	0	100
Slș.C 3	21.81	8.19	1.39	3.70	2.88	2.12	0.74	4.61	0.60	53.96	0	100
Slș.C 4	22.19	9.77	0.72	2.99	2.84	3.02	0.41	5.16	0.92	51.98	0	100
Slș.C 5	21.36	10.76	1.28	2.77	2.45	2.42	0.56	5.31	1.39	51.70	0	100
Slș.C 6	20.61	8.24	0.95	3.14	3.30	3.45	0.49	4.65	1.39	53.77	0	100
Slș.C 7	23.39	8.87	0.91	1.76	2.09	2.40	0.59	4.15	0.65	54.58	0.61	100
Slș.C 8	16.78	11.11	0.59	6.36	3.75	2.68	0.29	5.43	0.95	52.06	0	100
Slș.C 9	23.45	8.83	1.01	2.59	3.11	3.17	0.64	3.93	0.87	52.00	0.40	100
Slș.C 10	18.09	10.40	0.90	4.91	2.60	1.59	0.68	5.17	0.55	51.90	3.21	100
Slș.C 11	20.22	9.96	1.06	3.46	2.35	2.40	0.42	4.90	0.72	52.75	1.76	100
Slș.C 12	16.87	9.56	1.26	5.47	2.62	2.06	0.67	4.64	0.43	56.42	0	100
Slș.C 13	21.91	9.31	0.97	2.46	3.65	2.58	0.36	5.11	0.82	51.09	1.75	100
Slș.C 14	25.34	8.81	1.38	1.27	5.23	2.53	0.71	3.97	0.81	49.95	0	100
Slș.C 15	22.90	8.27	1.17	2.07	5.59	2.92	0.66	4.24	0.76	51.42	0	100
Slș.C 16	27.21	9.17	1.18	0.70	1.67	2.35	0.71	3.84	0.73	51.26	1.18	100
Slș.C 17	25.95	9.59	1.05	1.89	1.33	2.69	0.80	5.22	0.72	50.76	0	100
Slș.C 18	16.87	8.80	0.70	1.02	7.34	2.43	0.31	5.29	0.64	54.79	1.81	100
Slș.C 19	25.14	10.55	1.82	1.12	1.95	2.04	0.96	4.87	1.02	50.53	0	100
Slș.C 20	20.36	9.29	0.93	3.50	4.43	2.89	0.36	4.20	0.86	53.18	0	100
Slș.C 21	22.28	9.69	0.81	4.26	2.84	2.01	0.55	5.85	0.85	50.86	0	100
Slș.C 22	23.95	10.04	1.14	1.14	2.52	2.46	0.57	4.84	0.86	52.48	0	100
Slș.C 23	19.50	11.16	0.75	5.19	1.67	2.17	0.45	5.67	0.96	52.48	0	100
Slș.C 24	23.19	9.60	1.26	1.62	1.64	2.43	0.68	6.18	0.74	52.66	0	100

Therefore, the soil analyses will contribute significantly to the interpretation of the results obtained for the ceramic fragments. Archaeometric elements such as iron, calcium, phosphorus[20] and carbon have unitary concentrations (Table 2), which do not exceed 2% in the case of phosphorus and 3.73% in the case of calcium. The concentration of iron is relatively constant, between 4.15-5.80%, and that of carbon between 1.07-1.78%.

The iron concentration in the analyzed samples is equal to or exceeds the 4% in all fragments, suggesting the use of a ferruginous clay, with many iron oxides, visible in macro-

[20] BAKKEVIG, 1980, 73-100; KEELEY, 1981, 89-95; KSHIRSAGAR, 1991, 497–500; GOLYEVA *et al*., 2018, 313; SALISBURY, 2020, 199-211.

and microscopic analyses. Also, for three fragments (Slș.C14, Slș.C15, Slș.C18) the concentration of iron is exceeded by that of calcium, which reaches up to 7.33%, an aspect that may indicate the use of a calcareous clay[21] or the presence of carbonates. The origin of the calcium concentration will be investigated by µFTIR analysis.

Table 2. The elemental composition of the soil samples from Siliștea-*Pe Cetățuie* settlement (P1, P2-Cas.C 2018/2019; P3, P4-SIII2018/2019)

Elemental Composition in Weight Percent (%)

Soil Samples	Si	Al	Mg	P	Ca	K	Na	Fe	Ti	O	C	Total
Soil/P1	21.24	9.02	1.72	0.80	3.73	2.50	1.39	4.65	0.95	52.93	1.07	100
Soil/P2	22.05	6.67	1.30	0.57	3.72	1.65	1.02	4.45	0.45	56.34	1.78	100
Soil/P3	23.15	6.75	1.09	0.53	3.72	1.97	0.92	4.16	0.55	56.74	1.41	100
Soil/P4	23.38	6.72	1.15	0.62	3.55	1.88	1.00	5.80	0.67	53.92	1.31	100

The carbon is present in the paste of seven vessels (Slș.C7, Slș.C9-Slș.C11, Slș.C13, Slș.C16, Slș.C18) indicating a firing temperature that did not exceed 700°C[22], when this element disappears from the clay matrix. This aspect was also noticed from the SEM analysis of these fragments that have a low homogeneity and individualized mineral particles. However, the firing temperatures will be verified and completed by infrared spectroscopy analysis.

Phosphorus was identified in all fragments, but the 2%[23] limit was exceeded in 16 samples, that have values between 2.06-6.36%. In the category of cooking vessels, phosphorus is present in all four samples, with values of 2.23% (Slș.C1), 5.21% (Slș.C2), 3.69% (Slș.C3) and 2.90% (Slș.C4), thus supporting the use of pots in food preparation by boiling. Vessels intended for the preparation, serving and consumption of food contain, with one exception (Slș.C7), appreciable phosphorus concentrations starting from 2.59% (Slș.C9), 2.77% (Slș.C5), 3.14% (Slș.C5) .C6), 3.45% (Slș.1), 4.19% (Slș.C10) and reach up to 6.36% (Slș.C8), an aspect that suggests also the use of these vessels in the food preparation, most likely by boiling. Three storage solid or liquid goods vessels have in the ceramic paste a phosphorus content of 2.06% (Slș.C15), 2.46% (Slș.C13) and 5.47% (Slș.C12), which may suggest their use for food boiling, as well as for storage phosphorus-rich substances. Also, the storage of phosphorus-rich liquids, such as milk or wine, could also be done in vessels for storage or transportation of liquids, two of the samples from this functional class having an appreciable concentration of phosphorus, with values starting from 3.49 % (Slș.C20) and up to 5.19% (Slș.C23). A high concentration of phosphorus was also identified in the case of the multi-purpose vessel, where the value of this element is 4.26% (Slș.C21). Also, appreciable concentrations were identified in another study performed for

[21] MANIATIS, TITE, 1981, 61; RAVISANKAR *et al*., 2011, 374.

[22] PAPACHRISTODOULOU *et al*., 2006, 352; NODARI *et al*., 2007, 4669-4670; RAVISANKAR *et al.,* 2010a, 189; RAVISANKAR *et al.,* 2010b, 861; VASILACHE *et al*., 2020, 6.

[23] DUMA, 1972, 128; BÉARAT, 1994, 67; FREUDIGER-BONZON, 2005, 39; SANTOS RODRIGUES, LIMA DA COSTA*,* 2016, 297-298.

Costișa and Monteoru pottery from the Siliștea-*Pe Cetățuie* site, where important phosphorus content is present in the case of some preparation, serving and consumption vessels, in a storage pot, as well as on a multi-purpose container[24].

The EDX analysis of Costișa vessel surfaces illustrated the presence of elements specific to the raw material, but also elements resulted from the vessels use. For the containers that have a ceramic slip (Slș.C10, Slș.C11, Slș.C20, Slș.C22, Slș.C24) were not observed compositional differences that could indicate the use of other clays for these treatments.

Carbon is an important element that determines the interpretation of vessel functionality. Since for the Siliștea-*Pe Cetățuie* settlement we benefit from a series of compositional soil analyses, and the carbon concentration is on average 1.39%, we take into account the values which exceed 2% for the ceramic fragments, thus reducing the risk of contamination. Also, carbon values that are lower than 2%, but have traces of manganese, will be considered, the combination of elements indicating soot deposits[25]. Moreover, manganese was not identified in the soil samples analyses from the settlement, indicating the origin of this element from vessels use. Manganese has previously been found on the outer surface of three Monteoru vessels for storage solid or liquid goods[26].

Therefore, carbon is present on the outer surface of the Slș.C1 sample with values of 1.67%. Although the concentration is below the set limit, the existence of black deposits visible in SEM micrographs suggests the use of this vessel near a heat source. In the case of Slș.C4 sample, the 2.31% value of carbon was identified on the inner surface, indicating its origin from organic residues, idea also supported by a series of studies[27] that showed the fact that in over 80% of the cases, ceramic vessels contain organic residues absorbed in the paste.

Both samples (Slș.C1, Slș.C4) are part of the cooking category, and the results obtained through chemical analysis confirm the use of these vessels for food preparation with a heat source. From the category of preparation, serving and consumption vessels, carbon was identified only for the outer surface. The Slș.C5 sample has a carbon content of 10.63%, being also identified a stain deep in the ceramic surface, which has values of 39.12%, illustrating certain deposits caused by an organic fuel. The Slș.C7 sample has carbon values of 2.73%, and Slș.C11 has 0.40%, supplemented by 0.97% manganese, and thus, in this functional class, is highlighted the use of pots in food preparation with heat source. Also, the presence of carbon in association with manganese was identified in the category of solid or liquid goods storage vessels. These elements were identified on the samples Slș.C13 (2.32% C and 1.02% Mn) and Slș.C17 (0.37% C and 0.51% Mn), which show in the SEM micrographs black spots, indicating,

[24] BOLOHAN, 2013b, 41, 53/Annexes 7-8.

[25] ŁACIAK *et al.*, 2019, 427, fig. 4/g-l, 473.

[26] BOLOHAN, 2013b, 40, 53/Annexe 7.

[27] EVERSHED, 2008, 904.

also in this case, that some of the containers from this functional class could have been used to food preparation, most likely by boiling.

Scanning electron microscopy used to study the surfaces of Monteoru ceramic fragments, highlighted, as in the case of Costișa, three types of finishing (Fig.8). The vessels that have closed pores, mineral granules fully integrated in the paste with a smooth and compact appearance, present this treatment exclusively on the outer surfaces (Slș.M2E, Slș.M3E, Slș.M6E, Slș.M10E, Slș.M19E, Slș.M21E) a characteristic explainable by the accessibility to the external part of the pots. Coarse finishes give an irregular appearance, having individualized microgranules and are found mainly on inner surfaces that are more difficult to finish in the case of storage or transportation liquids vessels (Slș.M16E, Slș.M18E) or have been intentionally neglected in the case of preparation, serving and consumption vessels (Slș.M4I, Slș.M5I). The rest of the surfaces are semi-fine, well finished, relatively smooth, but with microgranules partially integrated in the clay matrix, being found in all functional classes. SEM micrographs for surfaces led to the identification of black spots on the outside of a preparation, serving and consumption vessel (Slș.M3E), representing a result of carbon deposits caused by the use of the container near fire[28].

Scanning electron microscopy performed on the Monteoru ceramic paste showed a good homogeneity, with mineral microstructural elements well integrated in the clay mass, indicating a firing at quite high temperatures (Fig.8). However, no vitrification[29] has been identified in any sample. A low homogeneity[30] was observed in seven samples (Slș.M5-M7, M13, M14, M19) where the mineral components are individualized, and in addition, in one sample (Slș.M16) were visible traces of burnt vegetal fibers. The compact lamellar structures with flattened pores, which indicate the use of the coiling technique, are visible in seven samples (Slș.M1, Slș.M2, Slș.M4, Slș.M5, Slș.M11, Slș.M20, Slș.M21), showing also at the microscopic level the use of this manufacturing pottery method.

As in the case of the Costișa, the chemical composition of the Monteoru samples contains the same elements specific to clay, such as aluminosilicates, quartz, feldspars, mica and iron oxides (Table 3).

Iron is present in all analysed samples with values between 3.83% and 11.35% indicating the use of a ferruginous clay[31] in vessels manufacturing. Also, as in the case of Costișa, were identified four samples that also have a high calcium content (Slș.M12, Slș.M13, Slș.M17, Slș.M21) with a maximum value of 6.91%, which may indicate the presence of calcareous clay[32] or carbonates.

[28] ŁACIAK *et al.*, 2019, 427, fig. 4/g-l, 473.

[29] MANIATIS, TITE, 1981, 61; KARAPUKAITYTĖ *et al.,* 2006, 386-387, Fig. 7-8; AMICONE *et al*., 2020, 14, Fig. 6/f-g.

[30] MANIATIS *et al.,* 1983, 777-778; PALANIVEL, MEYVEL, 2010, 340.

[31] MANIATIS, TITE, 1981, 61; NADEAU, TITE, 1987, 242-243.

[32] MANIATIS, TITE, 1981, 61; RAVISANKAE *et al*., 2011, 374.

The carbon is detected in seven samples (Slș.M5-Slș.M7, Slș.M13, Slș.M14, Slș.M16, Slș.M19) indicating a firing temperature lower than 700°C[33], aspect that will be clarified through μFTIR analysis.

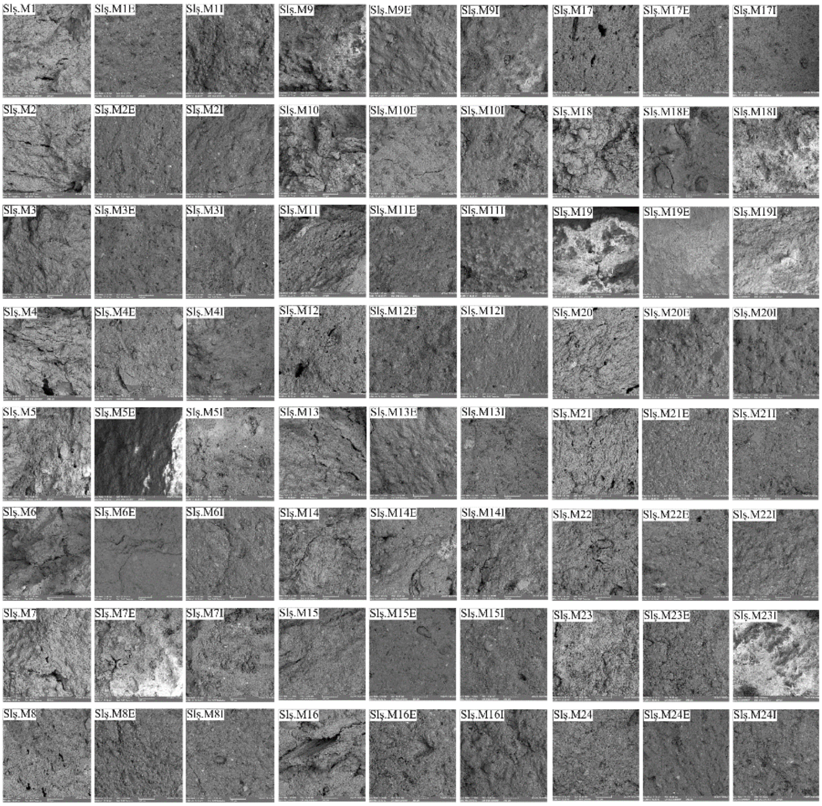

Fig. 8. SEM micrographs for Monteoru ceramic fragments (Slș.M1-Slș.M24) from Siliștea-*Pe Cetățuie* settlement (200× BSE)

Phosphorus concentrations exceed 2% in 15 samples, with values between 2.49-5.38%. These are present in the case of cooking vessels where they have values of 3.53% (Slș.M1) and 2.85% (Slș.M2) supporting the use of these pots in food preparation by boiling.

The same results were obtained for the vessels intended for preparation, serving and consumption of food, with the exception of Slș.M5, all other samples having phosphorus values of 2.59% (Slș.M3), 3.89% (Slș.M4), 4.27% (Slș.M7) and 5.38 % (Slș.M6). In the functional class of storage solid or liquid goods, are present, statistically, the most numerous representations of phosphorus. The values start from 2.49% (Slș.M14), 2.81% (Slș.M13), 3.08% (Slș.M11), 3.44% (Slș.M12), 4.24% (Slș.M9) and reach up to 5.28% (Slș.M15) suggesting, as in the case of Costișa, their use both in food preparation and in the storage of phosphorus-rich products. In the case

33 PAPADOPOULOU *et al.* 2006, 44; PAPACHRISTODOULOU *et al.,* 2006, 352; NODARI *et al.*, 2007, 4669-4670; BONG *et al.*, 2008, 304; RAVISANKAR *et al.*, 2010a, 189; RAVISANKAR *et al.*, 2010b, 861; AMICONE *et al.* 2020, 13; VASILACHE *et al.*, 2020, 7.

of vessels for storage or transportation of liquid, phosphorus was identified in two samples, having concentrations of 4.44% (Slș.M16) and 5.11% (Slș.M18). These values may be a result of using these containers to store liquids that are high in phosphorus, such as milk or wine. Also, this element was identified in the paste of a multi-purpose vessel, with a value of 4.99% (Slș.M22), indicating a possible cooking function of this container.

Table 3. The elemental composition of the Monteoru ceramic paste from Siliștea-*Pe Cetățuie* settlement

	Elemental Composition in Weight Percent (%)											
Monteoru Samples	Si	Al	Mg	P	Ca	K	Na	Fe	Ti	O	C	Total
Slș. M 1	24.59	9.26	1.32	3.53	3.06	2.59	0.71	3.90	0.75	50.29	0	100
Slș. M 2	23.79	9.63	0.97	2.85	2.36	2.62	0.53	4.24	0.73	52.28	0	100
Slș. M 3	25.26	9.78	1.38	2.59	3.63	3.00	0.66	4.36	0.71	48.63	0	100
Slș. M 4	23.33	9.42	1.01	3.90	3.04	2.45	0.53	4.37	0.76	51.19	0	100
Slș. M 5	15.56	7.74	1.39	1.55	2.78	1.72	0.94	4.14	0.68	59.51	3.99	100
Slș. M 6	17.17	10.86	1.13	5.38	2.99	2.33	0.61	4.82	0.82	52.64	1.25	100
Slș. M 7	18.86	8.97	0.75	4.27	3.00	2.44	0.40	5.16	0.80	54.60	0.75	100
Slș. M 8	25.72	9.62	1.30	1.55	2.31	2.70	0.69	4.27	0.67	51.17	0	100
Slș. M 9	20.38	9.79	2.12	4.24	2.72	2.42	0.63	4.30	0.70	52.70	0	100
Slș. M 10	24.53	9.57	1.30	1.13	2.14	2.63	0.79	4.45	0.67	52.79	0	100
Slș. M 11	18.82	8.34	1.34	3.08	3.19	2.47	1.00	11.35	0.55	49.86	0	100
Slș. M 12	16.75	8.52	1.11	3.45	6.15	2.32	0.27	6.49	0.94	54.00	0	100
Slș. M 13	15.36	9.07	1.23	2.82	5.05	3.21	0.67	5.11	0.74	54.26	2.48	100
Slș. M 14	23.47	9.28	1.48	2.49	2.85	3.09	0.73	4.56	0.77	51.15	0.13	100
Slș. M 15	17.26	11.11	1.52	5.28	2.86	1.74	0.47	5.33	1.02	53.41	0	100
Slș. M 16	22.61	8.67	1.53	4.44	3.47	1.76	0.53	6.48	1.26	48.24	1.01	100
Slș. M 17	25.10	9.47	1.91	0.78	6.92	2.72	0.70	3.83	0.58	47.99	0	100
Slș. M 18	19.62	9.59	0.78	5.12	3.37	2.57	0.47	4.25	0.99	53.24	0	100
Slș. M 19	21.13	9.82	1.44	1.74	1.05	2.00	0.82	4.35	0.58	56.83	0.24	100
Slș. M 20	25.76	8.89	0.96	1.08	2.46	2.40	0.44	4.94	0.77	52.30	0	100
Slș. M 21	22.03	9.87	1.70	1.56	5.43	2.43	0.66	4.77	0.93	50.62	0	100
Slș. M 22	20.41	10.23	1.15	4.99	3.77	2.59	0.51	4.80	0.71	50.84	0	100
Slș. M 23	22.76	10.15	1.47	1.70	2.17	2.60	0.60	4.90	0.64	53.01	0	100
Slș. M 24	24.17	10.35	1.08	1.06	1.95	3.20	0.59	4.87	1.09	51.64	0	100

The chemical composition of the surfaces of Monteoru vessels is specific to the raw material and the vessels that have a ceramic slip (Slș.M7, M8, M18, M21, M24) do not show significant compositional differences compared to the ceramic paste, indicating the use of the same clay.

Also, as in the case of Costișa, some elements with archaeometric value were highlighted on the surfaces of Monteoru pottery. Thus, carbon was identified on the exterior of three samples, having values from 2.50% (Slș.M1), 2.96% (Slș.M11) reaching 4.37% (Slș.M5), as well as on the inside of the Slș.M13 sample where its values are 2.20%. Carbon and manganese were identified on the outer surface of three samples, Slș.M2 (0.32% Mn), Slș.M3 (2.91% C and 0.56% Mn) and Slș.M14 (0.40% C and 0.63% Mn). These elements have been identified on the surfaces of cooking pots (Slș.M1, M2), preparation, serving and consumption vessels (Slș.M3, M5) and on storage solid or liquid goods containers (Slș.M11, M13, M14), used, like the Costișa ones, near fire.

B.3. µFTIR Analysis

The results obtained with the infrared spectroscopic analysis highlighted the existence of important similarities in terms of chemical compounds present in the 24 Costișa samples, being identified some exceptions that will be discussed in detail later (Fig.9).

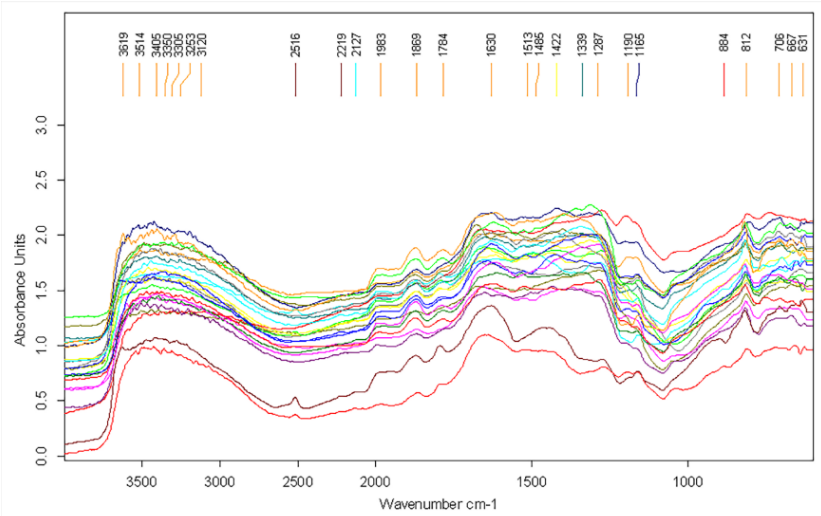

Fig. 9. FTIR spectra for the ceramic paste of the Costișa fragments from Siliștea-*Pe Cetățuie* settlement

The group of Costișa pottery fragments presents in the water region, between 4000-3000 cm^{-1}, peaks attributed to OH group (hydroxyl)[34] visible between 3405-3120 cm^{-1}, and OH deformations[35] at 1513 cm^{-1} and 1630 cm^{-1}, resulted from water absorption in the ceramic samples following the deposition processes or as a result of cleaning the ceramic fragments.

In all analysed samples, kaolinite[36] ($Al_2Si_2O_5(OH)_4$) was identified by the small peaks in the 3500-3750 cm^{-1} region. The absence of the ~3700 cm^{-1} peak[37] and that of the 915 cm^{-1} doublet[38] indicate that the vessels were fired at temperatures that exceeded 500-550°C.

34 AROKE *et al.*, 2013, 45; FORTE *et al.*, 2018, 135.

35 FROST, VASSALLO, 1996, 639; DAMJANOVIĆ *et al.*, 2011, 826; MANOHARAN *et al.*, 2015, 42; OANCEA *et al.*, 2017, 5085; COSTA *et al.*, 2017, 563.

36 FROST, VASSALLO, 1996, 639; TIRONIA *et al.*, 2012, 346; CHEN *et al.*, 2015, 30228; OANCEA *et al.*, 2017, 5085.

37 VELRAJ *et al.*, 2009, 731; PALANIVEL, KUMAR, 2011, 201-202; MANOHARAN *et al.*, 2015, 42; Velraj *et al.*, 2015, 936-937.

38 FROST, VASSALLO, 1996, 640; VELRAJ *et al.*, 2009, 731; RAVISANKAR *et al.*, 2010a, 189; MANOHARAN *et al.*, 2015, 42.

Intense peaks from 2928 cm^{-1} and 2861 cm^{-1} are attributed to organic carbon[39], present only on the inner surfaces of some vessels (Slș.C1, Slș.C3, Slș.C4, Slș.C7, Slș.C9, Slș.C10, Slș.C14, Slș.C19, Slș.C21). Organic carbon cannot be confused with organic matter which disappears after 250-300°C[40], when carbon dioxide is removed, so its presence is determined by the use of containers. The burning of organic matter was also highlighted by SEM analysis, where were visible only traces of vegetal fibers.

The intense peaks in 1300-1500 cm^{-1} region show the presence of carbonates[41], which indicates that the firing temperatures did not exceed 700-750°C[42], above this limit the calcite decompose[43] into gehlenite ($CaAl_2SiO_7$), diopside ($CaMgSi_2O_6$) and anorthite ($CaAl_2Si_2O_8$). Moreover, in two samples (Slș.C3, Slș.C18) the presence of calcite[44] was also noticed through the 2516 cm^{-1} and 884 cm^{-1} peaks. The different appearance of the two samples and the appreciable calcite content could indicate the use of another source of raw material in the manufacturing of these containers, aspect partially suggested by the EDX analysis. The lack of carbonates was noticed in the Slș.C11 and Slș.C24 samples, indicating higher firing temperatures.

The silicates are very well represented in the 2200-1870 cm^{-1} region attributed to the Si-O stretches[45] of quartz, and the intense peaks from 1983 cm^{-1} and 1869 cm^{-1} are visible in all samples. Furthermore, the presence of quartz[46] was also highlighted by the 2219 cm^{-1}, 2127 cm^{-1}, 1167 cm^{-1} and 1165 cm^{-1} peaks. Another silicate identified in all samples is muscovite[47] ($KAl_3Si_2O_{10}(OH)_2$), visible at 1190 cm^{-1} and 812 cm^{-1}. Diopside[48] ($CaMg[Si_2O_6]$) is present in all samples at the 631 cm^{-1} peak. These aluminosilicates are present in the raw clay used for the pottery manufacture, being also identified through mineralogical analysis.

Other siliceous minerals identified in all samples are represented by feldspars[49] at 1784 cm^{-1} and 1287 cm^{-1}, their presence being established by mineralogical investigations. Also, in all

39 COLUMBINI *et al.*, 2005, 85-36; MARITAN *et al*, 2005, 42; VELRAJ *et al.*, 2009, 731; DAMJANOVIĆ *et al.*, 2011, 862; KUMAR, RAJKUMAR, 2014, 35-36; VELRAJ *et al.*, 2015, 936; COSTA *et al.*, 2017, 573.

40 HÚLAN *et al.*, 2017, 83; ZUMAQUERO SILVERO *et al.*, 2020, 824.

41 NODARI *et al.*, 2007, 4669; RAVISANKAR *et al.*, 2010a, 187; BERZINA-CIMDINA, BORODAJENKO, 2012, 127.

42 MILLER, WILKINS, 1952, 1255; RAVISANKAR *et al.*, 2010a, 189, Table 2/2.

43 PAPACHRISTODOULOU *et al.*, 2006, 352; NODARI *et al.*, 2007, 4669-4670; RAVISANKAR *et al.*, 2010a, 189; RAVISANKAR *et al.*, 2010b, 861; VASILACHE *et al.*, 2020, 7.

44 KUMAR, RAJKUMAR, 2014, 32.

45 AROKE *et al.*, 2013, 49.

46 VELRAJ *et al.*, 2009, 731; NICULAE, 2011, 38, Table 5.1; PALANIVEL, KUMAR, 2011, 201-202; KUMAR, RAJKUMAR, 2014, 30-41, Table 2/1; CHEN *et al.*, 2015, 30228/Table.1.; VELRAJ *et al.*, 2015, 936-937; OANCEA *et al.*, 2017, 5085.

47 BARILARO *et al.*, 2008, 273, Table 1; VASILACHE *et al.*, 2020, 12.

48 NICULAE, 2011, 38, Table 5.1/16; OANCEA *et al.*, 2017, 5085.

49 OANCEA *et al.*, 2017, 5085.

spectra, the presence of iron oxides[50] was showed by the 667 cm^{-1} peaks, coming, like the other microstructural elements, from the raw material.

The FTIR spectra for the 24 Monteoru fragments have the same bend, showing the presence of the same chemical compounds in all samples (Fig.10). As in the case of the Costișa samples, the water region, between 4000-3000 cm^{-1}, is well highlighted by the 3424-3236 cm^{-1}, 1521 cm^{-1} and 1644 cm^{-1} peaks.

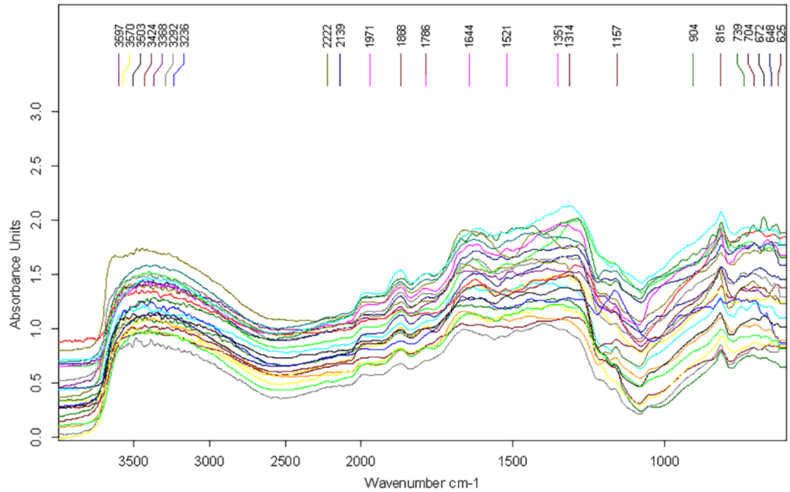

Fig. 10. FTIR spectra for the paste of Monteoru ceramic fragments from Siliștea-*Pe Cetățuie* settlement

In all analysed samples was identified the kaolinite[51] by the 3500-3750 cm^{-1} peaks, most of the samples being fired at temperatures above 500-550°C. In this regard, were identified two exceptions (Slș.M3, Slș.M12) where the presence of ~914 cm^{-1} the doublet[52] indicates a firing temperature lower than 500-550°C.

Also, in the case of some Monteoru samples, was identified the presence of organic carbon[53], through the small, but intense, peaks at 2948 cm^{-1} and 2856 cm^{-1}, visible on the inner surfaces of seven vessels (Slș.M3, Slș.M4, Slș.M10, Slș.M11, Slș.M12, Slș.M17, Slș.24).

The carbonates[54] are visible in all samples in the 1300-1500 cm^{-1} region, with one exception (Slș.M17), indicating firing temperatures below 700-750°C[55].

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50 RAVISANKAR *et al.*, 2010a, 188; NICULAE, 2011, 38, Table 5.1.

51 FROST, VASSALLO, 1996, 639; TIRONIA *et al.*, 2012, 346; CHEN *et al.*, 2015, 30228; OANCEA *et al.*, 2017, 5085.

52 FROST, VASSALLO, 1996, 640; VELRAJ *et al.*, 2009, 731; RAVISANKAR *et al.*, 2010a, 189; MANOHARAN *et al.*, 2015, 42.

53 COLUMBINI *et al.*, 2005, 85-36; MARITAN *et al*, 2005, 42; VELRAJ *et al.*, 2009, 731; DAMJANOVIĆ *et al.*, 2011, 862; KUMAR, RAJKUMAR, 2014, 35-36; VELRAJ *et al.*, 2015, 936; COSTA *et al.*, 2017, 57.

54 NODARI *et al.*, 2007, 4669; RAVISANKAR *et al.*, 2010a, 187; BERZINA-CIMDINA, BORODAJENKO, 2012, 127.

55 PAPACHRISTODOULOU *et al.*, 2006, 352; NODARI *et al.*, 2007, 4669-4670; BONG *et al.*, 2008, 304; RAVISANKAR *et al.*, 2010a, 189; RAVISANKAR *et al.*, 2010b, 861; AMICONE *et al.* 2020, 13; VASILACHE *et al.*, 2020, 7.

As in the case of the Costișa samples, the silicates are well represented in the specific region, between 2200-1870 cm^{-1}, and through the intense peaks from 1971 cm^{-1}, 1868 cm^{-1}, 2222 cm^{-1}, 2139 cm^{-1} and 1157 cm^{-1}. Other silicates identified are muscovite (815 cm^{-1}) and diopside (625 cm^{-1}). Feldspars were identified at 1786 cm^{-1}, 739 cm^{-1} and 672 cm^{-1}, and also in all the Monteoru fragments, are present iron oxides by the 648 cm^{-1} peak.

The SEM-EDX analyses performed enable to highlight some similar characteristics for the pottery of the two communities. The SEM analysis for the vessel's surfaces showed the presence of three finishing types, fine, semi-fine and coarse. Every one of them are found in all functional categories, both Costișa and Monteoru, aspect noticeable only at the microscopic level, reflecting partially the macroscopic observations on surface treatments. From a chemical point of view, no compositional differences were identified between the treatments applied to the surfaces and the paste, which suggests the use of the same type of clay in the preparation of the ceramic body and slip, a valid remark for the pottery of both communities. Thus, the homogeneity of the pasta is in most cases good, with a few exceptions. Also, through the scanning electron microscopy was confirmed the coiling technique of vessels manufacturing.

On the outer surfaces of some Costișa and Monteoru pots were identified carbon and manganese deposits. Thus, with the exception of cooking vessels, it appears that certain types of containers from other functional classes, such as those for preparing, serving and consumption and those for storage solid or liquid goods, could also be used for food heating or boiling. Traces of organic carbon were also discovered, showing the presence of absorbed residues in the ceramic paste as a result of vessels use.

The Costișa and Monteoru ceramic samples analysed through interdisciplinary methods have important compositional similarities, with some exceptions. Thus, the elements such as silicon, aluminum, potassium, magnesium and titanium identified by the EDX elemental analysis come from the aluminosilicates present in the FTIR spectra, which also represent a confirmation of the mineralogical observations. The analytical results of the Costișa and Monteoru samples showed that they were manufactured from a ferruginous clay. Regarding the identified exceptions, the Costișa Slș.C3 and Slș.C18 samples, they suggest the use of a calcareous clay, indicating as possible the usage of another source of raw material.

Phosphorus is present, in different amounts, on vessels of all functional categories, both Costișa and Monteoru. The identification of this element indicates the use of vessels for food preparation or for goods storage, either solid or liquid, that have a high phosphorus content, which argues and supports the proposed functionality of these containers. The high concentration of phosphorus in the EDX analyses and the lack of phosphates in the FTIR spectra, support the provenance of this element from the use of vessels. Moreover, the secondary firing spots, soot traces and carbon deposits on the surfaces of the pots indicates the use of these containers near fire.

By combining of the EDX with FTIR results was highlighted the presence of carbonates in almost all samples, indicating firing temperatures about 700-750°C. Moreover, for the Monteoru pottery were identified two samples that were fired at below 500-550°C, and other four samples (Slș.C11, Slș.C24, Slș.M6, Slș.M17) showed higher temperatures than 700-750°C. In this respect, the statistical representation of these temperature indicates some accidental exceptions, most likely caused by the vessels arrangement during firing and do not represent defining elements in determining temperatures ranges.

Therefore, through the EDX and μFTIR analyses, it was established the use of a local kaolinitic clay, with a high iron content, which has the same mineralogical and physicochemical characteristics. Regarding the elements of pyrotechnology, through interdisciplinary studies it was possible to determine that the pottery of both communities was generally fired at temperatures between 500/550°C-700/750°C. The various colors of the pots, the different firing atmosphere and the temperatures reached in this process show that the vessels firing was most likely done in open fire.

Conclusions

Based on the interdisciplinary analysis, it was possible to obtain important information about the Middle Bronze Age communities that made and used this pottery. The corroboration of the data obtained from the macroscopic and physicochemical analyses helped to partially reconstruct some ancient human behaviors related to the production and use of ceramic vessels.

The archaeometric analysis performed on the pottery from Siliștea-*Pe Cetățuie* settlement led to the identification of some general and particular aspects related to the ceramics of the two communities from here. The vessels of both ceramic groups were made using the same technique, being used mainly a local ferruginous clay extracted from the immediate proximity of the settlement. Moreover, ceramoclasts were used in both pottery assemblages, identifying three paste categories specific to certain functional classes, both for the Costișa and Monteoru vessels. Also, in the case of both communities were identified reused ceramoclasts, which could have a double role, one of a practical and technological nature, as well as a special one, of a socio-cultural nature.

The macroscopic and compositional results led to the confirmation of some vessels functionality, such as the cooking ones, as well as to the identification of multiple uses in the case of preparation, serving and consumption pots and of those for storage solid or liquid goods.

Regarding the pyrotechnology elements, through interdisciplinary studies it was established that the Costișa and Monteoru pottery from Siliștea-*Pe Cetățuie* settlement was fired at temperatures between 500/550°C-700/750°C. The fact that these temperatures do not correspond to certain types of vessels, the firing atmosphere is not uniform and the colors of the vessels are relatively different, suggests that the vessels of both communities were fired in pits or on ground.

To conclude, by using the interdisciplinary methods applied in this paper we were able to demonstrate that no notable differences were found between the pottery of the two communities. Manufacturing techniques, including the use of the same source of raw material, finishing treatments and firing technologies are the same, suggesting ordinary contacts between the Costișa and Monteoru ceramic groups in Siliștea-*Pe Cetățuie* settlement. Macro- and microscopic observations, pottery typology, physical characteristics and elementary compositions suggests the usage of the same technological skills, in order to produce the pottery needed by the Middle Bronze Age communities from Bistrița river's basin.

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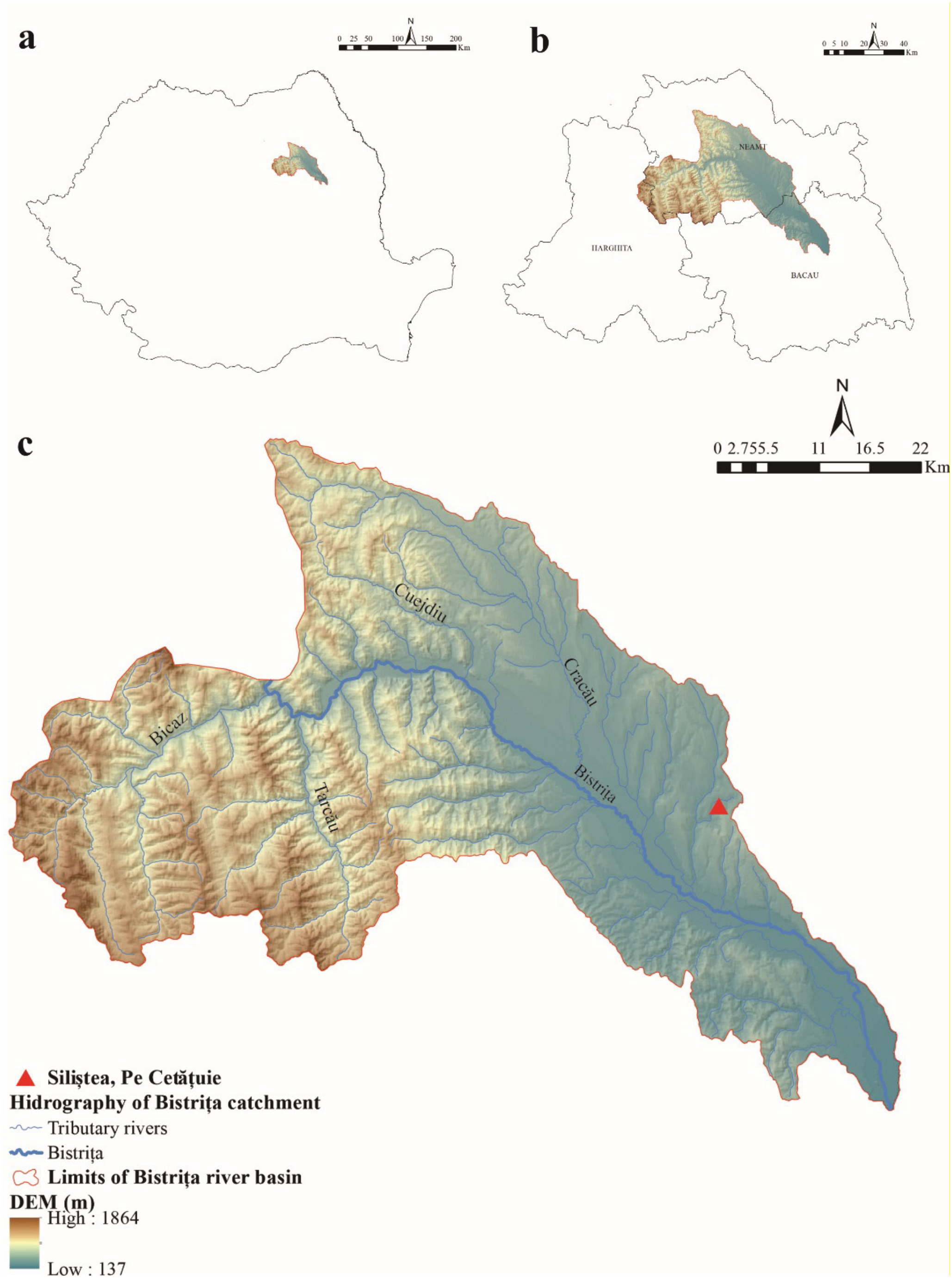
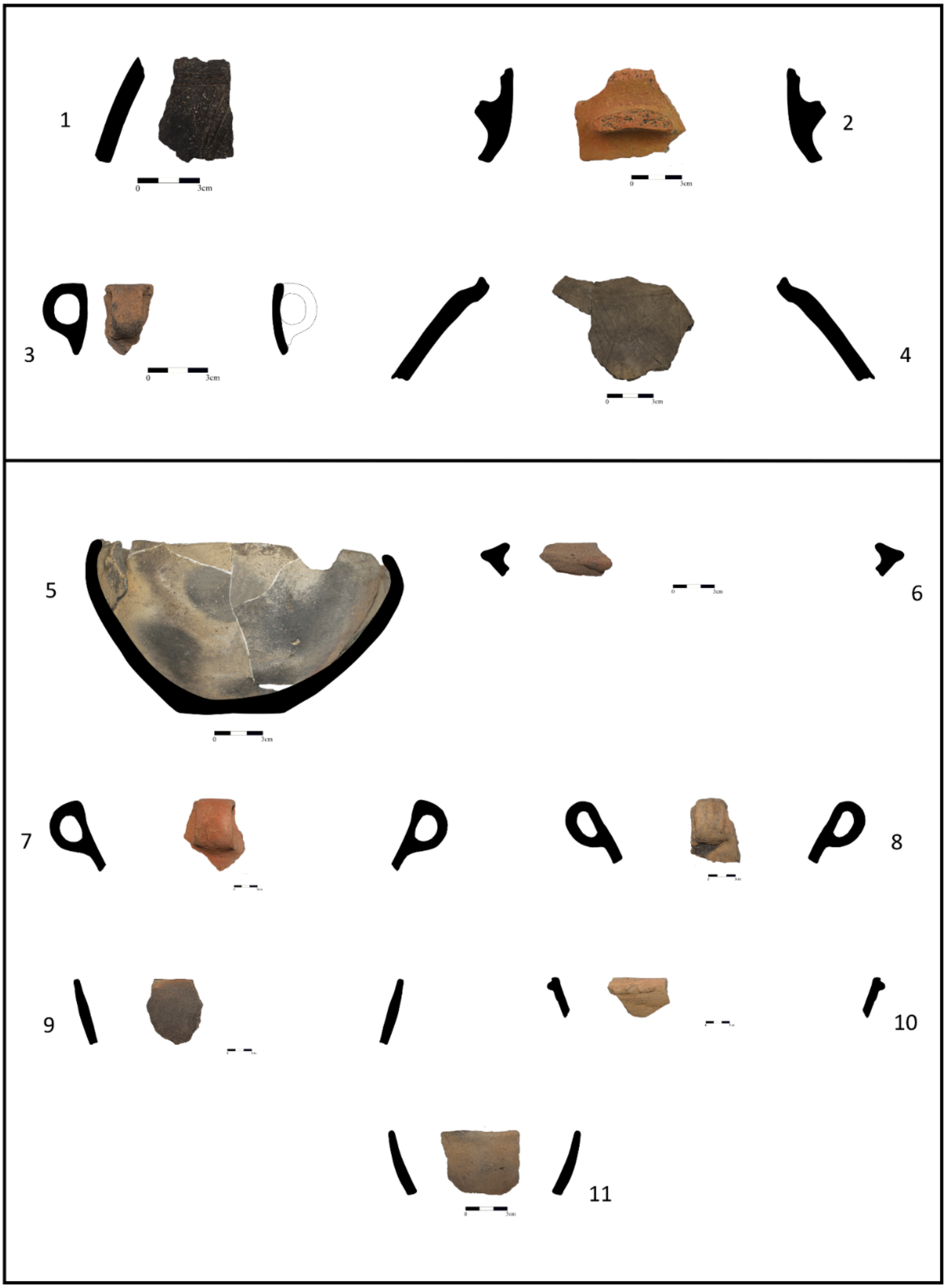
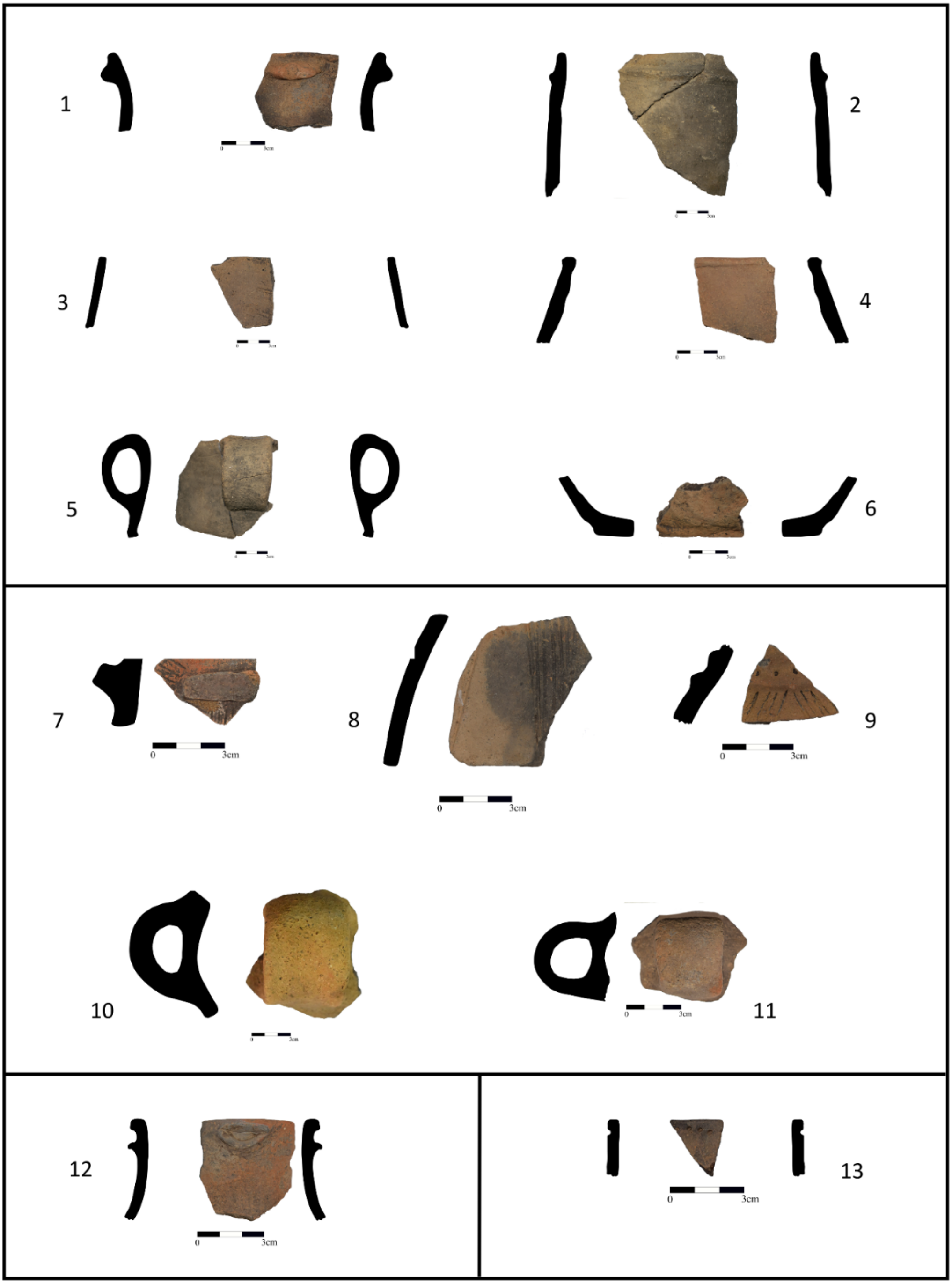


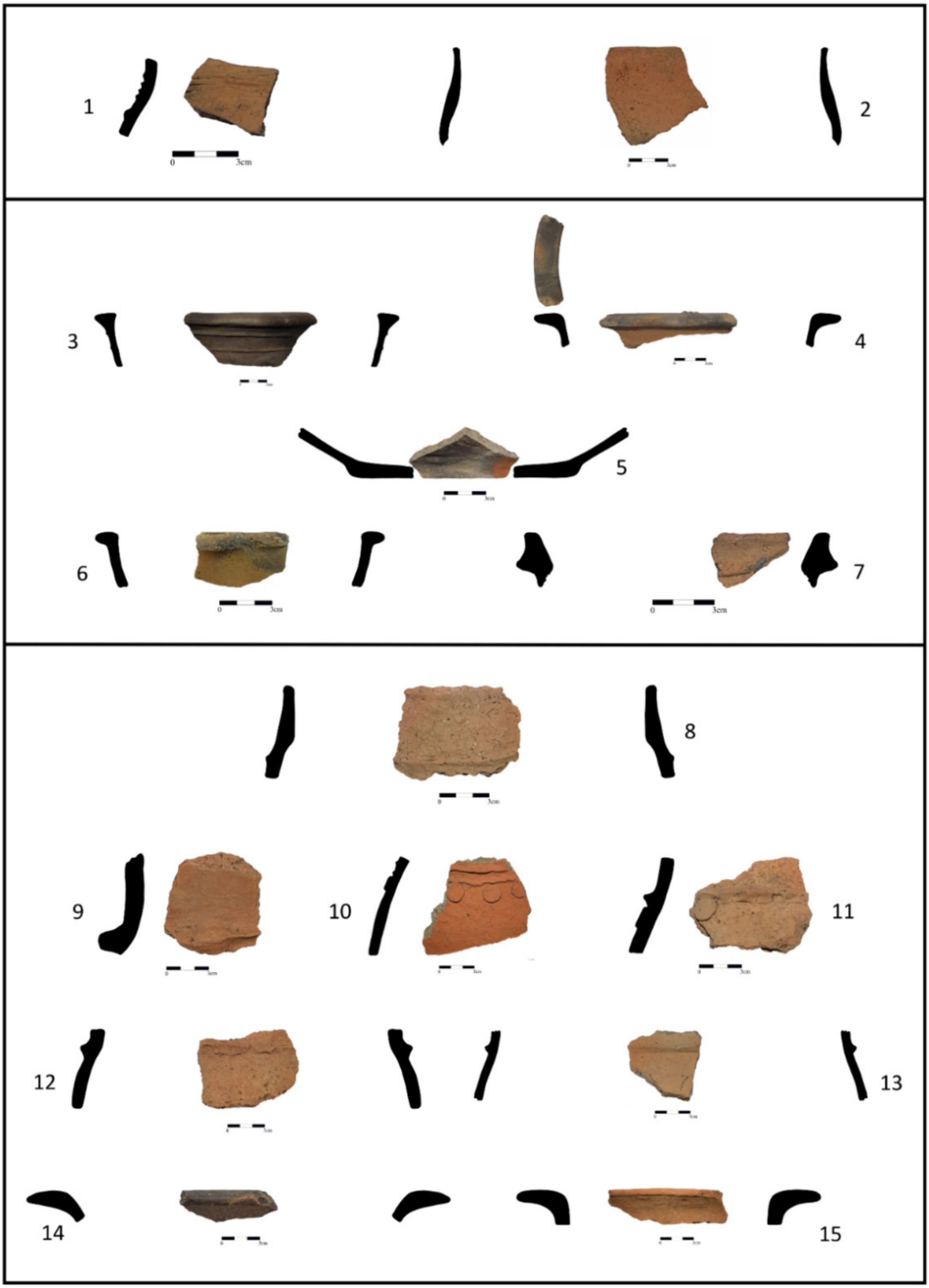
Map 1. Bistrița river's basin. a-Location on the territory of Romania; b-Position on the territories of Neamț, Bacău and Harghita Counties; c-Placement of Siliștea-*Pe Cetățuie* site on the Digital Elevation Model (SRTM 90, ARCGIS 10.6.1)



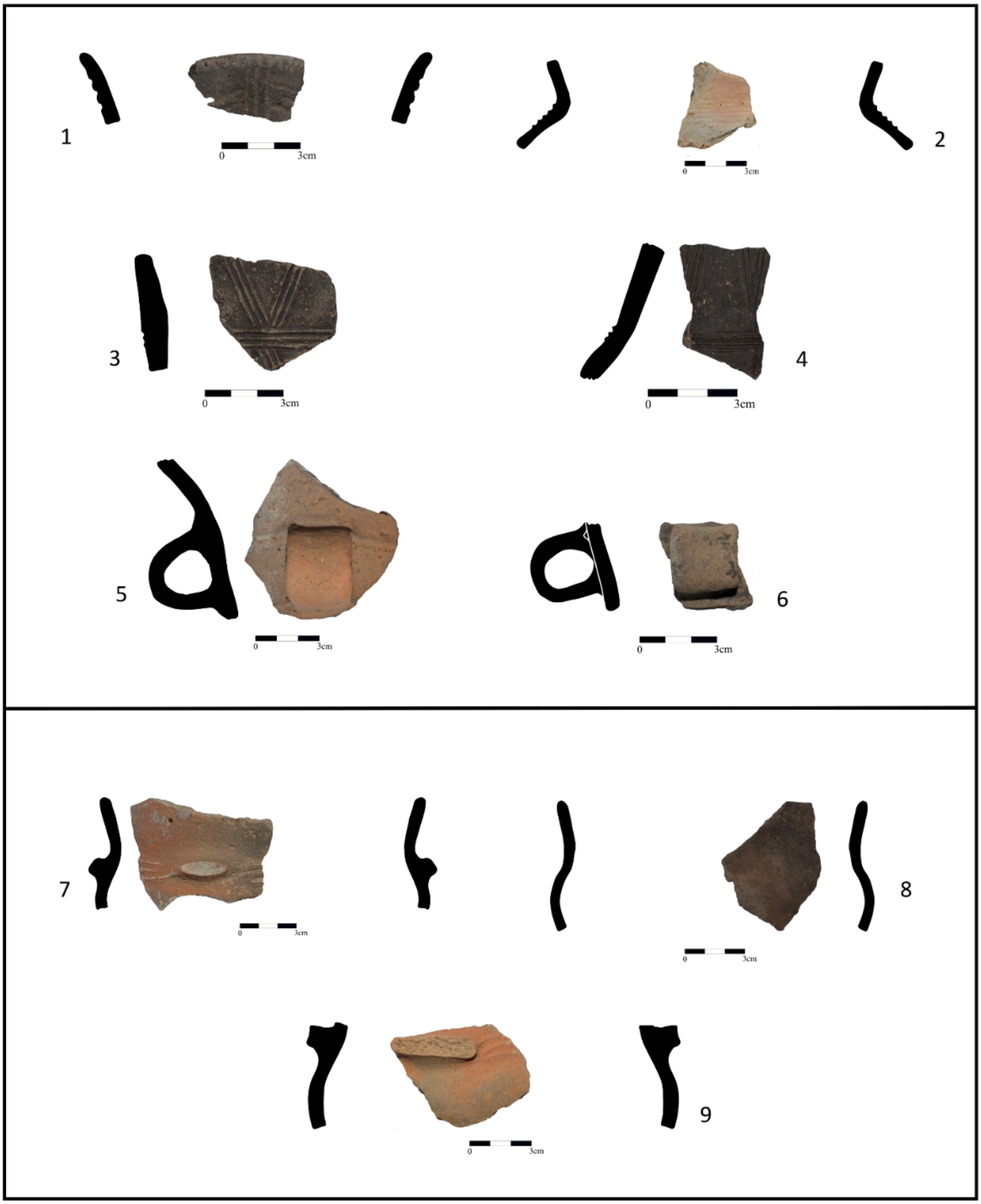
Pl. 1. Costișa pottery from Siliștea-*Pe Cetățuie* settlement: 1-4 Cooking vessels; 5-11 Preparation, serving and consumption vessels (Own archive images)



Pl. 2. Costișa pottery from Siliștea-*Pe Cetățuie* settlement: 1-6 Storage solid and liquid goods vessels; 7-11 Vessels for storage and transportation of liquids; 12 Special purpose vessel; 13 Multi-purpose vessel (Own archive images)



Pl. 3. Monteoru pottery from Siliștea-*Pe Cetățuie* settlement: 1-2 Cooking vessels;
3-7 Preparation, serving and consumption vessels;
9-15 Storage solid and liquid goods vessels (Own archive images)



Pl. 4. Monteoru pottery from Siliștea-*Pe Cetățuie* settlement: 1-6 Vessels for storage and transportation of liquids; 7-9 Multi-purpose vessels (Own archive images)