# Technological characteristics of the Cucuteni C pottery from Poduri–Dealul Ghindaru<sup>\*</sup>

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**Abstract**. The present work aims at investigating the technology used in the production of the Cucuteni C ceramic ware discovered at Poduri-Dealul Ghindaru archaeological site, with the help of a multidisciplinary approach making use of chemical and mineralogical analysis. The studied potsherds have shown the presence of different types of tempers, as determined by scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX) and X-ray diffraction (XRD). The type of tempers present within the ceramic matrix was identified by EDX and XRD analysis, while the dispersion of the different additives in the ceramic microstructure was determined by SEM measurements.

**Rezumat**. Prezentul studiu analizează tehnologia de realizare a ceramicii Cucuteni C din situl arheologic Poduri-Dealul Ghindaru, printr-o abordare multidisciplinară utilizată pentru determinarea compoziției chimice și mineralogice. Analizele au arătat prezența în pasta ceramicii a diferite tipuri de degresant, identificate prin microscopie electronică de baleiaj (SEM), analize dispersive de raze X (EDX) și difracție de raze X (XRD). Pentru identificarea tipului de degresant au fost realizate analize EDX și XRD, iar pentru determinarea microstructurii fragmentelor ceramice analize de microscopie electronică (SEM).

**Keyword**s: pottery technology, Cucuteni C, Poduri–*Dealul Ghindaru*, SEM-EDX analysis, XRD analysis.

## Introduction

The most abundant material identified within archaeological sites is ceramics, an important source of information for the study of prehistoric communities in terms of their technological knowledge, ancient trade patterns and social, cultural and economic

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complexity. A straightforward way to unravel some of the *technological skills* of the prehistoric communities and how different technologies were diffused and/or adopted is to analyse into more detail their ceramic artefacts.

In this context, mineralogical and chemical characterization of pottery may offer significant information about the provenance of the raw materials used<sup>5</sup> and also on the technology involved in the manufacturing process<sup>6</sup>. Additionally, important information regarding the techniques used to prepare the raw material (levigation, tempering with non-plastic materials and/or mixing of clays) and to assemble the vessel can be obtained by microscopically examining the shards<sup>7</sup>. The presence or absence of certain mineral phases provides information regarding the firing conditions (temperature and atmosphere) used during pottery production<sup>8</sup>. Further on, by analysing the firing process we can understand the clay mineral decomposition and reaction with the tempering minerals<sup>9</sup>.

Accordingly, the use of archaeometric methods for determining the pottery production technology can widely complement archaeological knowledge especially on its economic aspects which are related to the vessels function as well as on the entire production system.

Ceramic studies have occupied a forefront position in the archaeological analysis of the Cucuteni culture material remains since the discovery of the eponymous site in the 19<sup>th</sup> century. Much of the initial work related to Cucuteni ceramics focused on the development of typological sequences for decorated wares in order to develop relative chronologies.

Therefore, the archaeometric study of the Cucuteni pottery has focused mostly on determining the type of pigments used in the pottery production<sup>10</sup>. The pottery function<sup>11</sup> and the technological choices as determined by physical and chemical analyses<sup>12</sup> were investigated only recently.

<sup>&</sup>lt;sup>5</sup> VAUGHN, NEFF 2004, 1577–1586; ERAMO *et al.* 2004, 157–165; MARTINEAU *et al.* 2007, 23–52; SPATARO 2013, 175–197; RAMÓN, BELL 2013, 595–613; EERKENS, LIPO 2014, 25–31; CHIRIKURE *et al.* 2015, 23–32.

<sup>&</sup>lt;sup>6</sup> MANGONE et al. 2009, 97–102; GLIOZZO et al. 2008, 1074–1089; VŠIANSKÝ et al. 2014, 414–422; ÖZÇATAL et al. 2014, 2153–2160; JUNG et al. 2015, 455–463; ECKERT et al. 2015, 1–12.

<sup>&</sup>lt;sup>7</sup> TITE, MANIATIS 1975, 122–123; MENTESANA *et al.*, 2015.

<sup>&</sup>lt;sup>8</sup> RASMUSSEN et al. 2012, 1705–1716.

<sup>&</sup>lt;sup>9</sup> MAGGETTI 1982, 121–133; CULTRONE et al. 2001, 621–634.

<sup>&</sup>lt;sup>10</sup> CHIRIBUȚĂ 1979, 709–717; NICULESCU *et al.* 1982, 205–207; ELLIS 1984, 81–92; BURGHELEA *et al.* 2003, 251–258; CONSTANTINESCU *et al.* 2004, 125–134; 2007, 281–288; BUGOI *et al.* 2008, 195–199; BUZGAR *et al.* 2010a, 5–14; 2010b, 95–108; 2013, 2128–2135.

<sup>&</sup>lt;sup>11</sup> BODI 2010a, 127–183; 2010b, 199–210; 2015, 451–464; BODI, SOLCAN 2010, 315–323; MUNTEANU, GARVĂN 2015, 120– 122; MUNTEANU 2015.

<sup>&</sup>lt;sup>12</sup> CHIRIBUŢĂ 1979, 709–717; ELLIS 1980, 211–230; 1984, 157–159; GÂŢĂ 2000, 113–130; OLARU 2008; SANDU et al. 2010, 75–82; IONESCU, HOECK 2011, 937–958; 2012, 193–209; GRĂMĂTICU et al. 2010, 5–20; 2012, 229–246; MĂŢĂU et al. 2013, 914–925; 2014, 381–389; BODI 2015, 451–464.

This research is part of a more systematic archaeometric study focused on the Cucuteni C pottery<sup>13</sup>. In the previous investigation we took into consideration just a few samples which were analysed in comparison with the painted Cucuteni ware, especially in terms of firing technology<sup>14</sup>.

The present study aims to characterize the Cucuteni C pottery from Poduri–*Dealul Ghindaru* by determining its main technological features in order to assess the degree of variation in the Cucuteni C pottery production between sites. We present the characteristics of the raw materials used, and show a correlation between pottery composition and firing temperature, combining macroscopic examination with scanning electron microscopy with energy dispersive X-ray spectrometry (SEM-EDX) and X-ray diffraction (XRD). Our aim is not to address the origin of the Cucuteni C ware which is impossible to accomplish in one-step study without a systematic investigation of its chemical composition combined with clay analysis but to investigate its main technological parameters.

#### The archaeological context

The Cucuteni C pottery has interested scholars for more than eight decades because of its distinctness in terms of decorative patterns and paste composition in comparison with the painted Cucuteni ware<sup>15</sup>. Unlike Cucuteni A, A-B and B phases defined mainly based on changes in the painted pottery styles, the Cucuteni C has no chronological value<sup>16</sup>. C-type pottery existed for about 800 years, during Cucuteni  $A_3$ - $B_2$  stages (4300–3500 cal BC), being more frequent in the Cucuteni B sites<sup>17</sup>.

This pottery type was considered, based on its decorative style and paste composition (the use of shells as temper), to be a foreign element in the Cucuteni culture area. H. Schmidt argued for a Baltic origin for the Cucuteni C pottery fragments present in the upper strata of the B layer from Cucuteni–*Cetățuia* site<sup>18</sup>. Later on, archaeologists have considered these potsherds as belonging to ethnic groups originating in the Eastern Europe steppe region<sup>19</sup>. A. Dodd-Oprițescu in her systematic investigation which focused on identifying the "steppic" elements present in the Romanian Chalcolithic mentions the great variety of this ware type in

<sup>&</sup>lt;sup>13</sup> MĂŢĂU, MUNTEANU 2015; MUNTEANU, MĂŢĂU 2015; MĂŢĂU *et al.* 2015a; 2015b; 2015c.

<sup>&</sup>lt;sup>14</sup> MĂŢĂU et al. 2013, 914–925.

<sup>&</sup>lt;sup>15</sup> SCHMIDT 1932, 42–45; NESTOR, ZAHARIA 1968, 17–19; DODD-OPRIȚESCU 1977, 50–75; 1980, 547–557; 1981, 511–528; 1982, 70–79; CUCOȘ 1985, 63–92.

<sup>&</sup>lt;sup>16</sup> DODD-OPRIȚESCU 1977, 53.

<sup>&</sup>lt;sup>17</sup> DODD-OPRIŢESCU 1977, 63–68; CUCOŞ 1985, 63–92; for the Cucuteni culture chronology see: MANTU 1998, 111–133 and BEM 2001, 25–123.

<sup>&</sup>lt;sup>18</sup> SCHMIDT 1932, 42-45.

<sup>&</sup>lt;sup>19</sup> See DODD-OPRIŢESCU 1977, 50–53 with references therein.

terms of vessel shape and decoration during the same cultural phase<sup>20</sup>. Recent systematic analysis of the fire traces in relation with the vessel shape and the technological characteristics advanced the possible use as cooking pots for the Cucuteni C pottery<sup>21</sup>.

Because this ceramic is commonly believed to be a foreign element in the Cucuteni tableware it is important to determine its main technological characteristics before making any assumptions on its possible origin. Also, it became increasingly clear that there are significant regional and local variations in both form and techniques of production<sup>22</sup>. The history, nature and extent of these differences cannot be investigated based only on stylistic and typological analysis. All the archaeological hypotheses which considers the Cucuteni C pottery as being inferior in comparison with the painted ware need to be complemented by a multi-analytical approach, based on a combination of macroscopic, mineralogical, chemical and microscopic analysis of both temper and clay matrix.

The *tell* settlement from Poduri–*Dealul Ghindaru* is considered as a significant site for understanding the evolution of the Cucuteni culture (Figure 1). The multi-layered site is located in the Subcarpathian area, on the high terrace of the Tazlău Sărat River, being situated on its right bank.



Figure 1. Map showing the location of the Poduri–Dealul Ghindaru archaeological site.

<sup>&</sup>lt;sup>20</sup> DODD-OPRIŢESCU 1977, 73.

<sup>&</sup>lt;sup>21</sup> MUNTEANU, GARVĂN 2015, 122; MUNTEANU 2015.

<sup>&</sup>lt;sup>22</sup> DODD-OPRIȚESCU 1977, 73-79; CUCOȘ 1985, 63-92.

Recent non-invasive geophysical investigations suggests that the site was extend over around 6 ha<sup>23</sup>, being twice larger than previously considered<sup>24</sup>. The site is bordered by steep slopes on the northern and eastern side<sup>25</sup>. On the south-western side three ditches were revealed by the archaeomagnetic researches. However, no intrusive researches were conducted so far to demonstrate the relationship of these features with one or the other of the chalcolithic layers<sup>26</sup>. The *tell* was systematically excavated by different teams coordinated by D. Monah in 1979–1997 and 2000–2005 and by Gh. Dumitroaia during 2006–2009<sup>27</sup>. The anthropic deposit, more than 4.80 m thick, encompasses several Precucuteni and Cucuteni layers, starting from Precucuteni II until Cucuteni B<sub>2</sub>. Precucuteni III and Cucuteni A<sub>2</sub> phases are the best represented<sup>28</sup>. More detailed data are published from the later excavations which encompassed a Bronze Age layer and Cucuteni B and A<sub>2</sub> phases<sup>29</sup>. The inventory was discussed mainly in terms of *special finds* represented by painted pottery and anthropomorphic figurines.

At Poduri–*Dealul Ghindaru*, the Cucuteni C pottery has rather a scarce presence in comparison with the painted ware. D. Monah mentions its existence in the pottery assemblage even from the early excavations, assessing that there are no shells used as temper<sup>30</sup>. In the exhibition catalogue published in 2003, two Cucuteni C vessels are illustrated<sup>31</sup>, while in 2009 another five are reported<sup>32</sup>. In a systematic survey regarding the possible Cucuteni C pottery function, R. Munteanu mentions that 46 such vessels were retrieved between 1983 and 2009 at Poduri–*Dealul Ghindaru*. Just one of these is shell-tempered, the other 45 being attributed to C category based on their stylistic features<sup>33</sup>. Chronologically, the Cucuteni C pottery can be assigned broadly to the Cucuteni B phase.

## Materials and methods

The potsherds analysed here consists of 8 samples which were selected mostly from known stratigraphic units. Information regarding their microlocation and excavation year is

- <sup>29</sup> DUMITROAIA *et al.* 2009, 15–32.
- <sup>30</sup> MONAH et al. 1987, 13.

<sup>&</sup>lt;sup>23</sup> DUMITROAIA et al. 2012, 167, 173.

<sup>&</sup>lt;sup>24</sup> MONAH et al. 1980, 86.

<sup>&</sup>lt;sup>25</sup> MONAH et al. 1980, 86.

<sup>&</sup>lt;sup>26</sup> DUMITROAIA *et al.* 2012, 173.

<sup>&</sup>lt;sup>27</sup> DUMITROAIA *et al.* 2009, 11–13.

<sup>&</sup>lt;sup>28</sup> MONAH *et al.* 2003, 29–32.

<sup>&</sup>lt;sup>31</sup> MONAH *et al.* 2003, cat. no. 331, 374.

<sup>&</sup>lt;sup>32</sup> DUMITROAIA *et al.* 2009, cat. no. 25–27, 103, 182.

<sup>&</sup>lt;sup>33</sup> MUNTEANU 2015.

provided in Table 1. The pottery samples were selected to represent the typological and stylistic variability of the Cucuteni C pottery present at the site.

For information regarding the production technology, the selected pottery samples were studied by means of an integrated analytical approach combining macroscopic observation with chemical, mineralogical and microscopic investigations.

The macroscopic observation of ceramic pastes is very useful for preliminarily defining the technological and compositional variability of the pottery production. This approach can add important information on the different stages of the pottery production such as: paste preparation, firing conditions, manufacturing procedures and the surface finishing.

| Sample ID | Excavation<br>year | Excavation<br>unit                     | Sample | ₽  | Excavation<br>Year | Excavation<br>unit                    | Pottery<br>Sample |
|-----------|--------------------|--|--------|----|--------------------|---------------------------------------|-------------------|
| C1        | -                  | -                                      |        | C5 | 1982               | SIII,<br>□ 20 - 22                    |                   |
| C2        | 1984               | CAS A,<br>□ A -D,<br>-0.25 -0.35<br>cm |        | C6 | -                  | -                                     |                   |
| C3        | 2006               | CAS C,<br>□ C4,<br>-0.55-0.85<br>cm    |        | C7 | 1982               | SIII,<br>□ 20 - 22                    |                   |
| C4        | -                  | -                                      |        | C8 | 1987               | CAS A,<br>□ 7-10,<br>-0.80-0.90<br>cm |                   |

Table 1. The analysed pottery samples and their excavation context.

The chemical composition of all samples, in terms of major and some minor elements, and the microscopic analysis for detailed study of groundmass microstructure (low magnification) and the degree of vitrification (high magnification) was determined by Environmental Scanning Electron Microscopy – Energy Dispersive X-Ray (ESEM-EDX) analysis. The pottery fragments were sectioned and the resulting small sections were fixed on copper supports and their surface was examined using an Environmental Scanning Electron Microscope (ESEM) type Quanta 200, operating at 20 kV with secondary electrons in Low vacuum mode. The Quanta 200 microscope has an Energy Dispersive X-Ray (EDX) detection system for qualitative and quantitative analysis and elemental mapping.

The mineralogical composition was determined using a Shimadzu XRD 6000 diffractometer using CuK $\alpha$  radiation ( $\lambda$ =1.54059 Å) in reflection mode. A small quantity of each pottery sample (2 g) was powdered using an agate mortar and then side-pressed into a top-loaded holder in order to minimize the preferred orientation and analysed in the range of 2 $\theta$ =4° - 100° with a scan rate of 0.02° and 4s/step. Phase compositions were automatically identified by comparison with the reference powder patterns included in ICDD Powder Diffraction Files (PDF-4).

## **Results and discussion**

The macroscopic examination of the Cucuteni C pottery from Poduri–*Dealul Ghindaru* on the outer and inner surface revealed the existence of different colour shades which were determined by the adopted raw material and the variations in the atmosphere during firing (Figure 2). The colour of the outer surface of the selected samples varies from 7.5YR 5/3 (C1), 7.5YR 6/2 (C2), 7.5YR 6/3 (C7, C8), 7.5YR 7/3 (C3, C5) to 7.5YR 7/4 (C4, C6). For the cut-section of the Cucuteni C potsherds the colour ranges from 7.5YR 3/2 (C2), 7.5YR 4/2 (C5, C7, C8), 7.5YR 4/3 (C1) to 7.5YR 5/2 (C4). The colour spectrum for samples C1, C2, C4, C5, C7 and C8 is relatively uniform, while for samples C3 and C6 is more patchy varying from 7.5YR 7/4 to 7.5YR 5/3 (C3) and from 7.5YR 6/4 to 7.5YR 5/3 (C6).

All these differences in colours observed on the inner and outer surface and on the fresh sections are determined by the firing conditions which varies from inhomogeneous oxidizing with black core (C1, C3, C4, C5 and C7) to a more homogeneous oxidizing (C6) or reducing (C2 and C8). In the case of fragments exhibiting a black core and a lighter colour on the inner and outer surfaces the change in colour from core to margin is sharp for samples C5 and C7 and with more shaded boundaries for samples C1, C3 and C4. These samples corresponds to the so-called "sandwich structure"<sup>34</sup> which can be the result of a complex combination of oxidizing and redox conditions and, also, of the bulk chemical composition of the raw materials<sup>35</sup>.

<sup>&</sup>lt;sup>34</sup> NODARI *et al.* 2004, 119–128.

<sup>&</sup>lt;sup>35</sup> MARITAN *et al.* 2006, 1–15.



Figure 2. Macro-photograph and low-magnification micro-photograph (50×) of the vessel cut sections.



of the outer surface, matrix and temper of the Cucuteni C pottery fragments.

The macro-photograph and the low-magnification micro-photograph (50×) of the selected pottery samples, presented in Figure 2, indicate two main categories of Cucuteni C pottery: one containing bioclasts as temper (C1, C2, C5, C7 and C8) and the other having grog and quartz as temper (C3, C4 and C6). The bioclasts represented by shells fragments have a curved structure, sometimes with an observable lamination, being very frequent in samples C1, C5 and C7, while in sample C2 are smaller and less frequent (Figure 2). The abundance of grog and quartz inclusions, which are usually elongated and subangular, varies in sizes and quantities, being coarser in samples C3 and C6 in comparison with C4 potsherd.

In order to evaluate the degree of variability present in the chemical composition of the outer surface of the pottery fragments, their matrix and intentionally added temper we performed a multivariate statistical analysis using XLSTAT 2014 software. Hierarchical cluster analysis (HCA) enables to identify groups of objects within the data set.

The HCA analysis (Figure 3) revealed the presence of three main chemical groups within the Cucuteni C pottery from Poduri–*Dealul Ghindaru*. Shards belonging to group I are characterized by the shells used as tempering agent. The cluster analysis revealed the similarities between the outer surface chemical composition and between the shell fragments of the C2 and C8 samples which express a similar treatment of the outer surface and a similar composition of the additives. Sample C1, which is included in the same group, exhibits the existence of similar composition between the matrix and the temper. This may be the effect of the firing process which caused the disintegration of the shell structure and the increase of the CaO content also in the clay matrix.



Figure 4. Major element patterns of the outer surface of the pottery fragments.



Figure 5. Major element patterns of the pottery fragments matrix.



Figure 6. Major element patterns of the temper present in the pottery fragments.

Group II shows the similarities between the matrices of the grog tempered potsherds (C4 and C6) and of a shell tempered potsherd (C2) which can be the result of using a similar type of clay which was than processed with different type of additives and subjected to different firing protocols. The other pottery fragments included in this group are characterized by the presence of shells as intended additives. Also, the grouping of fragments which shares similar matrices (C7, C8), temper (C7, C5) and also outer surface (C1) compositions may be the result of the chemical reactions induced by the firing process.

The main characteristic of Group III is determined by the presence of samples which have a higher amount of  $SiO_2$  in the outer surface (C3, C4, C5, C6), in the matrix (C3 and C5) or in the temper (C3, C4 and C6). Another interesting feature of this group which reveals valuable information about the Cucuteni C pottery technology is represented by the existence of a similar matrix between a shell tempered pottery fragment (C5) and a grog tempered one (C3). C5 shell tempered pottery fragment shows also a similar chemical composition of the outer surface with the grog tempered fragments (C3, C4 and C6).

The spread between all the three groups of the shell tempered fragments in terms of pottery matrices composition reveals the existence of more than one recipe for the manufacturing of this pottery type. Also, the grouping of some of the grog and shell tempered fragments in terms of matrix chemical compositions shows that the archaeological separation based on visual examination does not corresponds to the existence of major differences between the different pottery types in all sequences of the *chaîne opèratoire* pottery processing.

With the aim of highlighting the features of the Cucuteni C pottery from Poduri–*Dealul Ghindaru* and, in the future, to integrate them in a more extend regional archaeometric Cucuteni C pottery analyses, the chemical composition of the outer surface, matrix and temper for the selected pottery samples was processed graphically on separate charts.

The major elements analysis for the outer surface of the analysed pottery fragments is presented in Figure 4. The data plotted in Figure 4 shows that the  $SiO_2$  varies from 32.94% (C1) to 65.16% (C6), the highest content being specific to the grog and quartz tempered pottery. The samples with the lowest  $SiO_2$  (32.94% for C1 and 44.84% for C8) have the highest content of CaO (19% for C1 and 11.19% for C8) which can be the effect of using a different clay source.

The high amount of  $Na_2O$  in sample C6 (3.67%) is possible to be determined by the salt added to the pottery paste or to the use of salty water in pottery manufacture.

In the calcareous clay, the use of salt may contribute to the delay of the calcination process and may prevent the appearance, in combination with  $H_2O$ , of calcium hydroxide (CaOH) which can deteriorate the pottery, being volumetrically greater than the initial CaCO<sub>3</sub> and  $H_2O^{36}$ . The chemical composition of sample C6 exhibits rather a non-calcareous clay,

<sup>&</sup>lt;sup>36</sup> HAMDAN et al. 2014, 997.

having the lesser amount of CaO (1.05%). A possible origin of the high  $Na_2O$  present in sample C6 may relate to the feldspar content which is the result of a variation in the production recipe, or of the pottery alteration after its production or during the post-depositional process<sup>37</sup>.

The  $P_2O_5$  content is less than 1% in six of the analysed pottery samples, while in C4 is 1.44% and 3.05% in sample C5. High concentration of  $P_2O_5$  can be influenced by the ancient content and/or post-depositional process<sup>38</sup>. This is determined by the exchange reaction of phosphorous between soil and shards takes place at the surface of the pottery vessel, at vesicles or at least at the phase boundaries<sup>39</sup>. For the high amount of  $P_2O_5$  in the outer surface of samples C4 and C5, we infer a rather post-depositional origin.

The major element analysis for the pottery matrix of the analysed samples is shown in Figure 5. The  $SiO_2$  and CaO content exhibits almost the same pattern as for the outer surface chemical composition, except for sample C5 which has the highest  $SiO_2$  content (74.47%) from all the samples.

The dataset presented in Figure 5 can be correlated with the one from Figure 3 which shows a similar grouping for C5 and C3. The similar grouping pattern observed for samples C5 and C3 can be assign to the use of a similar type of clay in the pottery production. Also, sample C5 has the lowest values for all the other major elements. The content is more homogeneous and has lower concentrations than in the outer surface. The very similar  $P_2O_5$  concentrations mean that the core surface was not affected by post-depositional contamination.

The major element composition for the temper used in pottery production is given in Figure 6. The data presented in Figure 6 are more scattered than the initial grouping in shell tempered and grog and quartz tempered pottery as defined by macroscopic observation. The shell tempered pottery shows a greater variation in the  $SiO_2$  and CaO concentrations when compared with the grog and quartz group which is caused by reaction with the clay matrix during the firing process.

In the grog and quartz temper group, sample C4 has the highest content of  $Na_2O$  which corresponds to the increase in the Na-feldspar (albite) during the firing process.

The  $P_2O_5$  content shows increased values, especially, in the pottery fragments with bioclasts as temper. The presence of  $P_2O_5$  in the temper cannot be assigned to the vessel content but was rather formed during the firing process by the partial melt of the organic temper<sup>40</sup>.

<sup>&</sup>lt;sup>37</sup> BUXEDA I GARRIGÓS et al. 2002, 9.

<sup>&</sup>lt;sup>38</sup> KLEIN *et al.* 2004 with the references therein.

<sup>&</sup>lt;sup>39</sup> KLEIN et al. 2004, 350.

<sup>&</sup>lt;sup>40</sup> KLEIN *et al.* 2004, 350.

In view of the matrix homogeneity of the studied samples, based on the variations observed in the chemical composition, it is possible to assume that the slight differences among the identified two main groups (shell tempered and grog and quartz tempered) could be related to the heterogeneous features of the initial clays or, alternatively, to the use of different raw materials.

Micro-morphological analyses by scanning electron microscopy allows estimation of the textural characteristics of groundmass and temper, offering information on the transformation reached by the samples during the firing process. When the micro-morphological analyses are combined with the mineralogical information we can obtain a rough estimation of the equivalent firing temperatures.



Figure 7. Microphotograph showing the microstructure of the C1, C2, C3, and C4 pottery fragments.

The results of the microscopic analyses for samples C1, C2, C3 and C4 are presented in Figure 7, while Figure 8 contains the results for samples C5, C6, C7 and C8.

SEM microphotographs exhibit the differences in microstructure between the two groups: the shell tempered and the grog and quartz one. These differences developed during firing consist in the appearance of new mineral phases in the clay matrix or at the boundaries between clay matrix and the used temper<sup>41</sup>.



Figure 8. Microphotograph showing the microstructure of the C5, C6, C7 and C8 pottery fragments.

<sup>&</sup>lt;sup>41</sup> RICCARDI *et al.* 1999, 393–409.

The microstructural characteristics of the groundmass for the shell tempered samples show similar characteristics in terms of pore distribution as can be observed for samples C1, C5, C7 and C8 (Figure 7 and 8). Sample C2 (Figure 7) groundmass presents some differences in the distribution and pore sizes in comparison with the other samples containing shells as temper. These differences are determined by the firing reducing atmosphere.

As regards the microstructural changes due to the firing process, the shell fragments used as additives exhibits some variety. Shells present in samples C1, C5 and C8 (Figure 7 and 8) have a lamellar structure and are characterized by the appearance of intra- and inter-layer pores. The appearance of inter-layer pores can be caused by the decomposition of the organic compounds present in the shell structural elements or to the calcite decomposition<sup>42</sup>.

Based on the presence of intra-layer pores, we may consider that the firing temperature does not exceeded  $800^{\circ}C^{43}$ . The shell fragments present in sample C2 (Figure 7) are much smaller and embedded in the clay matrix making difficult to assess their microstructural transformations. In sample C7 (Figure 8), the shell fragment has a prismatic morphology which is related to the differences in shell species<sup>44</sup>. Based on the reduced number of pores and on their location between adjacent layers, but also inside the layers we can assume that the firing temperature was in the range of 750–800°C<sup>45</sup>.

SEM study of the grog and quartz tempered pottery fragments enabled us to determine some differences between the samples. Textural features revealed two types of tempers: a *sandy* grog associated with larger quartz grains (samples C3 and C4 — Figure 7) and a *carbonate* grog (sample C6 — Figure 8). The carbonate grog present in sample C6 (Figure 8) presents a matrix which varies from sandy to clayish<sup>46</sup>. Samples C3 and C4 (Figure 7) display large grains of quartz which show no grain-to-grain contact with the matrix. All three samples show no sign of vitrification which enable us to consider that the firing temperature did not exceeded  $800°C^{47}$ .

The mineralogical composition as determined by XRD analyses is shown in Figure 9. The major minerals which are present in all the analysed pottery samples (Figure 9) are quartz and calcite. Micas and clay minerals (illite), feldspars and plagioclase are the minor phases in the ceramics, while hematite and magnetite are present as trace phases.

XRD patterns of the Cucuteni C pottery revealed the presence of illite/muscovite in all the pottery samples. The existence of the phyllosilicate minerals indicates that the firing

<sup>&</sup>lt;sup>42</sup> MARITAN *et al.* 2007, 536.

<sup>&</sup>lt;sup>43</sup> MARITAN *et al.* 2007, 537.

<sup>&</sup>lt;sup>44</sup> ESTEBAN-DELGADO et al. 2008, 153–165; GÉNIO et al. 2012, 86–103.

<sup>&</sup>lt;sup>45</sup> MARITAN *et al.* 2007, 535–536.

<sup>&</sup>lt;sup>46</sup> ZULUAGA *et al.* 2011, 446.

<sup>&</sup>lt;sup>47</sup> MANIATIS, TITE 1981, 61.



p-plagioclase, f-feldspar, m-magnetite, h-hematite).

temperature did not exceed  $850-900^{\circ}C^{48}$ . In samples C1, C5, C7 and C8, due to the high amount of shell tempers, the calcite reflection prevails. As a result of lowering the temper quantity, sample C2 is not dominated by the calcite peak.

The grog and quartz tempered group (samples C3, C4 and C6) are dominated by the quartz reflection. Moreover calcite dominant peak is not present and the other calcite peaks have lower intensity. Additionally, plagioclase peaks are more intense, while alkali feldspars (f) have rather a sparse intensity.

Hematite and magnetite are present in samples C2, C3, C4, and C6, but with peaks of lower intensity. The formation of hematite and magnetite depend on the firing atmosphere prevalent during the manufacturing process. The presence of weak intensity peak of

<sup>&</sup>lt;sup>48</sup> DAMJANOVIĆ *et al.* 2011, 825.

magnetite is determined by the transformations of  $Fe_3O_4$  to  $Fe_2O_3$  during the firing process<sup>49</sup>. Samples C3, C4 and C6 exhibit a pale ununiformed orange colour which is determined by the alternation of oxidative and redox condition during the pottery firing<sup>50</sup>.

The association of both hematite and magnetite in the pottery C3, C4 and C6 potsherds reveals that samples are fired under changing conditions established from the ununiformed colour range. On the other hand sample C2 which has both hematite and magnetite exhibits a black colour which is the result of the carbon formed during reduced atmospheric firing using charcoal and wood<sup>51</sup>.

These observations can be correlated with the results of the chemical composition HCA analysis. Moreover, they show the same pattern in the mineralogical composition as the one in the chemical composition of C2 sample with the grog and quartz tempered group. This trend suggests the use of a similar clay source for the matrix composition, even if they were subjected to different tempering and firing process.

XRD data analysis can also provide some insights into the production process used to manufacture the Cucuteni C pottery. The appearance of phases such as hematite and magnetite in samples C2, C3, C4 and C6 suggests that the firing temperature attained  $850^{\circ}$ C, while the maintenance of illite/muscovite enable us to consider that the temperature did not exceed  $900^{\circ}$ C<sup>52</sup>.

## Conclusions

A multi-analytical approach to the investigation of the Cucuteni C pottery excavated at the archaeological site of Poduri–*Dealul Ghindaru* enabled us to gain insight into its production technology. By determining the microscopic characteristics, the chemical and mineralogical composition it was possible to clearly determine the similarities and differences between the two groups determined macroscopically. In addition, the investigation of pottery production process clearly indicates the *technological choices* made by potters regarding raw material selection, paste recipes and firing process<sup>53</sup>

As a group, the shell tempered Cucuteni C pottery show general similarities in textural characteristics, mineralogical and chemical composition. Compared to the grog tempered Cucuteni C pottery, the shell tempered samples have a higher CaO content and a lower  $SiO_{2,}$  and with the exception of C2 sample show now traces of newly formed mineral phases.

<sup>&</sup>lt;sup>49</sup> PALANIVEL, KUMAR 2011, 59.

<sup>&</sup>lt;sup>50</sup> NODARI *et al.* 2007, 4665.

<sup>&</sup>lt;sup>51</sup> MANOHARAN *et al.* 2015, 45.

<sup>&</sup>lt;sup>52</sup> DAMJANOVIĆ *et al.* 2011, 825.

<sup>&</sup>lt;sup>53</sup> SILAR, TITE 2000, 2–20.

The grog and quartz tempered Cucuteni C ware exhibit a higher content of  $SiO_2$  and small amounts of CaO. Their mineralogical composition shows the existence of hematite and magnetite as newly formed phases, alkali feldspars and plagioclase as minor phases. The similarity in terms of chemical composition between the grog tempers and the pottery matrices in which they are embedded allow us to consider that previous potteries was worked with clay from the same source area.

While differences between shell and grog-quartz tempered pottery from Poduri–*Dealul Ghindaru* are quite apparent, equally striking are the similarities between the two, especially in matrices composition and morphology. Nevertheless, the variability in the chemical data seems to point to the use of a non-standardized recipe in the paste preparation.

Physicochemical characterization methods therefore provide experimental evidence which can help us to understand the Cucuteni culture pottery production technology and to clarify the initial archaeological hypothesis. Much more work is clearly needed, but the results achieved to date shed new insights on the production and technology of the Cucuteni C pottery. Finally, more detailed archaeological and archaeometric analyses are needed for explaining the timing, tempo and significance of the Cucuteni C pottery in the whole area of the Cucuteni-Trypillia civilization.

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