Observations at microscope on pottery fabric of some ceramic fragments from Giumelnița tell settlements Hârșova and Bordușani Popină

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Abstract: In this paper we present preliminary results of microscopic study on pottery fabric of few ceramic fragments discovered in Chalcolithic tell settlements Hârșova and Bordușani Popină. Analysis of 20 sherds considered the description of their paste fabric in terms of texture, microstructure, porosity, composition and colour, in order to characterize technological issues related to preparing the mixture and combustion conditions, identify materials used, and indicate possible natural sources for them. The main identified paste types are characterized by the presence, constantly, of small pottery fragments ("crushed ceramic"), as well as plant fragments such as straw, or even coprolites (probably sheep and goat types, as suggesting the presence of phyto-sferolites). A special case is that of the bivalve shell fragments that are observed not only in ceramic fragments attributed to Cernavodă I culture (as presented in literature), but also in one of Giumelnița ceramic fragments from Bordușani, as with the fine carbonate fragments (limestone rounded grains). Also were found units of surface finishing (fine pure clay slip) added at the external surface of pots, and simple surface (mostly external) finish in very wet state, without addition of clay. Combustion temperature is estimated quite low because not produce transformations of mineral grains and does not allow the complete combustion of vegetable matter in the mixture. The ceramic fragments from Hârșova present oxidation zone in the external part, while some ceramic fragments from Bordușani present symmetrical zones of oxidation, suggesting different ways of arranging the dishes in the oven (for example, for Hârșova in stack, upside down). To complete this study, it is particularly important to perform the clay mineral analysis by X-ray diffraction method. Also, this study must be developed in a systematic way, corresponding to the main types of paste that can be recognized macroscopically and with binocular magnifier, and also by their correlation with the types of vessels identified.

Rezumat: În acest articol prezentăm rezultate preliminare ale studiului la microscop asupra pastei unor fragmente ceramice descoperite în așezările eneolitice de tip tell Hârșova și Bordușani Popină. Analiza celor 20 de fragmente ceramice a avut în vedere caracterizarea organizării pastei acestora din punct de vedere textural, al microstructurii, porozității, compoziției și culorii, în scopul caracterizării aspectelor tehnologice referitoare la prepararea amestecului și condițiile de ardere, identificării materialelor utilizate și indicarea posibilelor surse naturale pentru acestea. Principalele tipuri de pastă identificate sunt caracterizate prin prezența, în mod constant, a fragmentelor de ceramică de mici dimensiuni („cioburi pisate”), ca și a fragmentelor vegetale de tipul păilor, sau chiar a coprolitelor (probabil de ovicaprine, după cum sugerează prezența fito-sferolitelor). Un caz special este acela al fragmentelor de cochilii de bivalve, ce sunt observate nu numai în fragmentele ceramice atribuite culturii Cernavodă I (cum este prezentat în literatură de specialitate), dar și pentru unul dintre fragmentele ceramice giumelinitene de la Bordușani, ca și în cazul fragmentelor carbonatice fine (granule de calcar rotunjite). De asemenea, au fost întâlnite unități de finisare a suprafeței (slip de argilă fină, pură) adăugate la exteriorul vaselor, cât și simpla finisare a suprafeței (cel mai adesea externă), în stare foarte umedă, fără adăos de argilă. Temperatura de ardere este estimată ca una destul de scăzută, deoarece nu produce transformări ale granulelor minerale și nu permite arderea completă a materiei vegetale din amestec. Fragmentele ceramice de la Hârșova prezintă zone de oxidare numai în partea externă, în timp ce unele dintre fragmentele ceramice de la Bordușani prezintă zone de oxidare simetrice, ceea ce sugerează modul diferit de dispunere a vaselor în cuptor (de exemplu, la Hârșova, în stivă, cu gura în jos). Pentru completarea acestui studiu, este deosebit de importantă analiza materialelor argiloase prin metoda difracției de raze X. De asemenea, acest studiu trebuie dezvoltat într-o manieră sistematică, corespunzător principalelor tipuri de pastă, ce pot fi recunoscute macroscopic și la lupă binoculară, ca și corelarea acestora cu tipurile de vase identificate.

Keywords: Chalcolithic, Giumelnița, pottery fabric, microscopic study.

Cuvinte cheie: Eneolitic, Giumelnița, pasta ceramicii, studiu microscopic.

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1. Preliminary considerations

Fabric analysis of pottery is the study of the features related to fine (clay) matrix, inclusions and firing temperature and conditions (C. Orton et alii 1993, p. 67).

Ceramic paste is the subject of detailed chemical and mineralogical analysis in order to characterize technological aspects related to the composition of mixture (matrix and inclusions), and identify the materials used, as well as natural sources for them. One of the most commonly used methods for the study of ceramic constituents is the petrographic analysis (D. Şeclăman et alii 2003, p 200). If identifying sediments in thin section under the microscope is done considering optical and mineralogical criteria, analyzing the microstructure, porosity and organic inclusions envisages the micromorphological criteria allowing characterization of materials prepared by mixing such: bricks, adobe, mortar, etc, as well as the transformations suffered by them (M.-A. Courty et alii 1989, p 72-74). The overall microstructure, the porosity and the birefringence fabrics, are very important features providing clues on the modality of preparing the ceramic paste (C. Hâită 2003, p. 79-80).

Nature of inclusions can distinguish the types of ceramic paste from different cultural areas, or coming from different settlements, in relation to available materials. Identification of organic matter in ceramic paste, as well as the transformations experienced by some mineral grains (such as clay minerals and quartz; D. Şeclăman et alii 2003, p. 201), may indicate the temperature interval of pottery burning.

In what follows, we intend to present the results of microscopic analysis on the ceramic fabric, on issues related to the preparation of the paste of ceramic fragments, as well as the different materials used, without having detailed information on natural sources, as complementary mineralogical and chemical analysis were not available.

2. Material and method

For the purpose of this study twenty pottery fragments were analyzed in thin section; these ceramic samples are coming from the micromorphological thin sections made on archaeological occupational units investigated in the tell sites from Hârşova (15) and Borduşani (5), which are attributed to cultural levels Gumelniţa A2 – 18 and Cernavodă – 2 (only from Hârşova). Because most sections are covered with glass slides, it was not possible to perform chemical analysis by physical methods. Also, this study do not intends the characterization of all different types of paste that can be observed macroscopically or with binocular magnifier, for all kind of pottery found in two sites, but intends to present some preliminary observations, which will be developed in a systematic study.

The microscope study is performed in the laboratory of the National Center of Pluridisciplinary Researches in Bucharest, using an Olympus BX 60 petrographic microscope, in polarized light (PPL and XPL), at magnifications between x100 and x500, and the photographs were done using the Olympus analogical camera adapted for this microscope.

Microscopic analysis took into account all the characteristics that are analyzed for the prepared materials such as adobe, clay, mortar, plaster and other materials made by mixing. These features are: texture, microstructure, porosity, composition and colour (C. Hâită 2003, p. 68).

Textural features indicate the types of sediments used, how they were sampled and the presence of any intentional mixing of sediments, in order to increase non-plastic granular content (degreaser) in clay. The sorting degree and the maximum size of sedimentary grains are the most important parameters to be considered here in order to understand if the mineral inclusions were intentionally added as tempering material or occur naturally in the clays. Some clays could be washed to remove the very coarse inclusions before they can be used (F.R. Matson 1971, p. 594).

Microstructure expresses the degree of homogeneity of the mixture, the general distribution of the materials (non-preferential or preferential arrangement, outlined by the existence of zones or bands) and clay orientation relative to coarser grains (shown in birefringence fabric). It provides information on the modality of the paste preparation and the subsequent transformations by combustion.

Porosity is characterized by isolated circular voids formed by preparing mixture in wet state (air bubbles trapped in clay) or voids and channels resulted by burning organic matter added to the mixture, and through fissures formed by separation of different areas of material and cracking during firing, resulted by the rapidly elimination of water.

Overall composition of the ceramic paste concerns both fundamental mass of mineral constituents - clay or other fine sediments (fine matrix), and mineral or organic compounds added as
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degreasing (inclusions) - grains of quartz, limestone, shell fragments, ceramic fragments, fragments of clay or daub, plant fragments, such as straw and husks, coprolites, etc. The mineralogical analysis of the sedimentary matrix indicates its source (wind deposits – loess, alluvial, lacustrine, etc.). Chemical and petrographic analysis of added constituents characterizes the type of paste and its physical characteristics. Inclusions added as degreaser are described by size, frequency, shape, roundness and sorting.

The colour expresses the overall composition, the nature of constituents (especially iron compounds and organic matter) and the distribution thereof (discrete inclusions or clay mass oxides), but also ceramic combustion conditions (temperature, duration and atmosphere combustion). Although is very important that the description of colour is done using a standard chart, this is difficult to assess under microscope; also the colour is variable, under the firing conditions.

The dark core of the ceramics can be used as a means of the analysis of the degree of low-temperature firing. To estimate the degree of combustion, it must be considered that the temperature and the duration of combustion are inter-dependent variables and texture and porosity directly affects the degree of transformation of clay paste. The dark core is thus the expression of the "depth" to which the thermal transformation is done. In the case, the pottery was fired insufficiently with respect to both temperature and duration to eliminate this darker zone, and usually is meaning that a kiln was not used (F.R. Matson 1971, p. 595).

Also, very important is to observe the colour differences between surfaces and margins, resulting from conditions after burning, by cooling (O.S. Rye 1981, p. 116; C. Orton et alii 1993, p. 69). In this case, for oxidizing firing, there are not significant changes (O.S. Rye 1981, p. 117). Other thermal transformations could result by secondary burning, if the potteries come from structures destroyed by firing.

3. Preliminary results of the analysis at the microscope

3. 1. Texture
All ceramic fragments analyzed have very fine textured sedimentary matrix consisting of clay, containing rare and fine non-plastic grains. They are represented mainly by grains of mono- and polycrystalline quartz, feldspar and micas (pl. I/fig. 2, 3). In terms of their frequency and size were identified the following types:

1. Very fine fabric with clay texture (pl. I/fig. 1);
2. Fine fabric with clayey silt texture; in this case, the silt grains of 20-40 microns in size, and rarely fine sand, 63-100 microns in size, have frequencies of 3-5% and 5-10% rarely; in this case, the sedimentary matrix presents a good sorting (pl. I/fig. 2);
3. Medium (semi-fine) fabric with clayey silt and fine sand texture, which, along with silt grains, fine sand grains are observed with sizes from 63 to 200 microns, rare of 200-250 microns, which have usually frequencies of 10-20%, and in one case approximately 30% (pl. I/fig. 3); the sedimentary matrix has a moderate sorting and the grain size distribution has a bimodal aspect; in some cases the fine sand grains implies a heterogeneity of the paste (zones of concentration).

These textural features indicate that there is no mixing of sediments, only different ways of sampling and, possibly, of preparation. Individualization of the three textural types would indicate that there is likely a better control on sediment texture used for making ceramic paste of the fragments analyzed. A single ceramic piece with the content of about 30% fine sand granules with the maximum size of 250 microns; in this case, the sedimentary material has a moderate sorting, suggesting a possible mixing during sampling from alluvial sequences.

Also, these textural variations were observed in the case of the ceramic fragments ("crushed ceramics") included in the paste.

3. 2. Microstructure
Ceramic fragments studied have, in general, a homogeneous microstructure, indicating a good mixture preparation. The presence of coarse grains (pottery, limestone granules, and fragments of shells) defines microstructure of porphyric type (pl. I/fig. 4), in their absence, ceramic fragments analyzed have a massive microstructure.

For the finishing units such as slips, a banded microstructure is characteristic (pl. I/fig. 5). There were observed both situations, the case where slip is applied externally, generally consisting of fine and pure clay (pl. I/fig. 6), and where no clay is added for the wall finish (pl. II/fig. 1).
The birefringence\(^1\) fabrics indicate the orientation of clay minerals around coarse grains added as a degreaser (pl. II/fig. 2) and striated fabric, parallel to the surface of vessel. A very good orientation of clay is observed for the finishing zones as slip, where the pure fine clay was applied in a very wet state.

### 3. 3. Porosity
Paste porosity of analyzed pottery fragments can be classified into two distinct categories. In the first case, there are only isolated circular pores, determined by the air trapped in the sediment at the time of preparation (pl. II/fig. 3), or circular pores and fine channels, obtained by burning plant material added to the mixture (pl. II/fig. 4).

In the second category there are the fine fissures made by firing, parallel to the surfaces. Generally, the cracks are very fine, <1 mm, but frequent. They are very common in the case of the very fine fabric, when vegetable matter was added, where millimeters cracks are interconnected and associated with voids that include charred vegetable fragments (pl. II/fig. 5).

### 3. 4. Composition
For the majority of ceramic fragments analyzed, inclusions are represented by fine fragments of pottery (“crushed ceramics”) and plant fragments. The pottery grains have sizes between 0.2 and 2 mm, rarely 2.5-3 mm, angular shapes and low frequency, 10-20\%, and rarely 30-40\% (pl. I/fig. 3; 4; pl. II/fig. 2). Were observed several instances where ceramic fragments include, in turn, other pottery (pl. II/fig. 6), indicating the reuse of these materials; the ceramic fragments of a sample have different texture and combustion transformation degree. Accidentally, in the composition, granules of clay or daub were noted. They are more common in the pottery fragments assigned stratigraphically to the Cernavodă I cultural level from Hârșova.

Rarely are encountered rare millimeter-sized fragments of bivalve shells (pl. III/fig. 1) and carbonate grains (pl. III/fig. 2). Shell fragments are characteristic of ceramic fragments attributed to Cernavodă I culture, as described in the literature, but were observed also for a pottery fragment from Bordușani, attributed to A2 phase of Gumelnita culture.

Plant fragments are present in very variable proportions and characterize generally the very fine and fine fabric types. There are situations in which decayed plant fragments are observed - vegetables that may become either from coprolites, either from organic clayey sediments.

A special case is represented by the two fragments attributed to Cernavodă I culture, in which plant material is very frequent (about 40-50\%) and includes common vegetable debris. Also, dense organic aggregates with fibrous microstructure and rounded morphology were observed, resulting most likely from the sheep and goats coprolites (pl. III/fig. 3). In some situations were observed phyto-sferolites of calcium oxalate (pl. III/fig. 4), specific (M.-A. Courty et alii 1989, p. 114, pl. III/f) for the waste and coprolites of this category of animals, that also are more compact, and include strongly disturbed phytoliths and vegetable debris.

### 3. 5. Colour
Regarding the colour of studied ceramic fragments, it should be noted that there are considerable differences between the external wall, the internal wall and the central zone (pl. III/fig. 5). In general, the external zone has colours in shades of red to yellow reddish indicating an oxidative combustion, while the inner part, light brown - dark brown, suggesting a predominantly reducing combustion ambiance, which might suggest that the vessels were burned either upside down or stacked one above another.

Individualization of oxidation zones indicates that the combustion is made in dominant oxidizing conditions. The vast majority of the ceramic fragments have a brown-dark brown internal zone, containing vegetable matter, which is not transformed sufficiently to be destroyed by burning, in some cases recognizing plant structure (pl. III/fig. 6).

The differences between some pottery fragments from Hârșova and from Bordușani are related to the conditions of firing. Possibly this indicates the different arranging of pots in the firing structure.

The oxidant character of the surface may indicate a rapid cooling in open air or “the opening” of the oven before cooling. There are situations when, for the same type of paste, colour is variable, which might indicate a relatively low temperature control.

\(^1\) The birefringence is the optical feature of different minerals corresponding to XPL polarized light vibration on the two optical axes in thin section.
4. Conclusions

Microscopic analysis of these ceramic fragments showed the presence of three main types of paste, identified primarily by the texture of sedimentary matrix. These three types are typically associated with different types of inclusions:
- very fine textured fabric contains only vegetable fragments;
- fine sandy textured fabric may not contain vegetable matter when the frequency of sand granules reaches 20-30%;
- the fabric with fragments of shells, although characteristic of ceramic fragments attributed Cernavodă I culture, is observed very rarely - in one case of Gumelnita ceramics from Borduşani. Here, very rarely are observed also carbonate granules.

Textural characteristics of sediments used, including the observed variations and also the mineralogical nature of fine sand and silt fractions, indicate a local source, located most likely in areas where fine alluviums of the Danube were available for sampling. The same could be indicated by the constant presence of very fine altered plant fragments that can derive from natural sediments.

Although for ceramic fragments assigned to Cernavodă I culture were observed fine calcitic grains, there are not arguments supporting in any way that sediment source could be represented by deposits of loess, as for the main types of construction materials of these tell settlements, prepared by mixing with plant material - daub.

Regarding microstructure, it was observed that ceramic fragments attributed to Cernavodă I culture have a strong heterogeneity in the central part and a high content in vegetable matter. In the cases studied, it was observed a very thin slip of pure clay, the clay particles being very well oriented, parallel to the vessel walls. A systematic study may indicate that there are different “recipes” for preparing, or other elements of production technology of ceramic paste.

Regarding burning, it is very clear that the duration and temperature of combustion were not sufficient for complete combustion of vegetable matter included in the paste. The same is indicated by the presence of dark core, which occur when, under the action of temperature, equilibrium is not established for the entire thickness of the vessel wall. On the other hand, there are not notable changes in the minerals constituent, this indicating clearly a thermal domain below the 700˚C. The degree of fine fraction birefringence, which represents the optical response of clay minerals determined by individual particles orientation, also indicates a low temperature combustion, which did not cause important transformations of these minerals. The presence of organic amorphous compounds indicate a short combustion at a temperature of 300-400˚C (D. Ţedăman et alii 2003, p. 203). Such situations are presented in the literature as a result of short-term burning in an oxidizing atmosphere. The same situation was observed for the ceramics from the Starčevo-Criş site of Foeni-Şalăş site (M. Spataro 2003, p. 40). The low firing temperature was sufficient for making pottery good for its aims, and also required less fuel. The very dark, organic core, of Cernavodă I pottery is explained by the high organic content and possibly dung (probably from sheep and goat animals) content.

Greater frequency of cracks, as almost complete burning of vegetable matter and a red uniform section may indicate in some cases of the very fine fabric ceramic fragments, burning at a higher temperature, but as well, the fact that tempering was not good enough, the interconnected fissured suggesting that the pottery obtained was not very adequate as liquids container.

Based on currently available data, we can say that the ceramic paste from the two Gumelnita settlements have very similar characteristics. As for the pottery attributed to Cernavodă I culture is evident that presents distinct characteristics, whose specificities must be detailed in a systematic study.

To complete this study, it is particularly important to perform the clay mineral analysis by XRD method and by other quantitative analysis. Mineralogical analysis of silt and sand fractions may indicate possibly different sampling sites of sedimentary material.

Also, this study must be developed in a systematic manner, by reference to the main types of paste that can be identified macroscopically and at the stereomicroscope, and by correlation of the results with the types of vessels identified.
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Fig. 1. Pottery fragment with very fine texture and well developed fissures. XPL, f. w. 2 mm. Fragment ceramic cu textură foarte fină și fisurație bine dezvoltată. XPL, l. i. 2 mm.

Fig. 2. Pottery fragment with fine texture, including a vegetable fragment. PPL, f. w. 2 mm. Fragment ceramic cu textură fină ce include un fragment vegetal. PPL, l. i. 2 mm.

Fig. 3. Pottery fragment with fine sand moderate texture, including pottery fragments. XPL, f. w. 2 mm. Fragment ceramic cu textură medie, cu nisip fin, ce include fragmente ceramice. XPL, l. i. 2 mm.

Fig. 4. Pottery fragment with fine texture, with frequent pottery fragments. XPL, f. w. 2 mm. Fragment ceramic cu textură fină, cu frecvente fragmente ceramice. XPL, l. i. 2 mm.

Fig. 5. Pottery fragment with banded microstructure, including vegetable fragments. PPL, f. i. 2 mm. Fragment ceramic cu microstructură în benzi, ce include fragmente vegetale. PPL, l. i. 2 mm.

Fig. 6. Pottery fragment with fine clayey slip, including vegetable fragments. XPL, f. w. 2 mm. Fragment ceramic, cu slip argilos fin, ce include fragmente vegetale. XPL, l. i. 2 mm.

Pl. I. Photos at the microscope. PPL – plan polarized light; XPL – Crossed polarized light; f. w. – frame width. Imagini la microscop. PPL – lumină plan polarizată; XPL – lumină polarizată încrucișat; l. i. – lățime imagine.
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**Fig. 1.** Pottery fragment with wall polishing unit with pure clay. XPL, f. w. 2 mm.
Fragment ceramic cu finisare a peretelui cu argilă fină. XPL, l. i. 2 mm.

**Fig. 2.** Pottery fragment showing birefringence fabric. XPL, f. w. 2 mm.
Fragment ceramic ce prezintă microstructură de birefrinjență. XPL, l. i. 2 mm.

**Fig. 3.** Pottery fragment with vesicular porosity, with isolated voids. XPL, f. w. 2 mm.
Fragment ceramic cu porozitate veziculară, cu pori izolați. XPL, l. i. 2 mm.

**Fig. 4.** Pottery fragment with vesicular and channeled porosity. PPL, f. w. 2 mm.
Fragment ceramic cu porozitate veziculară și cu canale. PPL, l. i. 2 mm.

**Fig. 5.** Pottery fragment with frequent interconnected fissures and vegetable debris. XPL, f. w. 2 mm.
Fragment ceramic cu frecvente fisuri interconectate și resturi vegetale. XPL, l. i. 2 mm.

**Fig. 6.** Pottery fragment including another pottery fragment with crushed ceramics. XPL, f. w. 2 mm.
Fragment ceramic ce include un alt fragment ceramic cu ceramică pisată. XPL, l. i. 2 mm.

**Pl. II.** Photos at the microscope. PPL – plan polarized light; XPL – Crossed polarized light; f. w. – frame width.
Imagini la microscop. PPL – lumină plan polarizată; XPL – lumină polarizată încrucișată; l. i. – lățime imagine.
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Fig. 1. Pottery fragment including a shell fragment. XPL, f. w. 2 mm. 
Fragment ceramic ce include un fragment de cochiile de bivalve. XPL, l. i. 2 mm.

Fig. 2. Pottery fragment including a carbonate granule. XPL, f. w. 2 mm. 
Fragment ceramic ce include un granul carbonatic. XPL, l. i. 2 mm.

Fig. 3. Pottery fragment including a coprolite fragment. XPL, f. w. 2 mm. 
Fragment ceramic ce include un granul coprolitic. XPL, l. i. 1 mm.

Fig. 4. Pottery fragment with coprolite aggregate with vegetable debris and phyto-sferolites. XPL, f. w. 2 mm. 
Fragment ceramic cu agregat coprolitic cu debris-uri vegetale și fito-sferolite. XPL, l. i. 1 mm.

Fig. 5. Pottery fragment with fine clay slip and very organic internal zone. XPL, f. w. 2 mm. 
Fragment ceramic cu slip argilos fin și zonă internă foarte organică. XPL, l. i. 2 mm.

Fig. 6. Pottery fragment including a vegetable structure. PPL, f. w. 1 mm. 
Fragment ceramic ce conține o structură vegetală. PPL, l. i. 1 mm.

Pl. III. Photos at the microscope. PPL – plan polarized light; XPL – Crossed polarized light; f. w. – frame width. 
Imagini la microscop. PPL – lumină plan polarizată; XPL – lumină polarizată încrucișată; f. w. – lățime imagine.