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Dedicated to Professor Liviu Literat
On the occasion of his 85th birthday

MINERALOGICAL AND PHYSICAL CHARACTERISTICS OF ROMAN CERAMICS FROM HISTRIA, *BASILICA EXTRA MUROS* SECTOR, WEST-EAST SECTION (ROMANIA)

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ABSTRACT. The paper presents the results of the mineralogical and physical investigation performed on 10 fragments of Roman ceramics (oil lamps, bowls, plates) out of the 20 samples collected from the west-east Section of the *Basilica extra muros* Sector, Histria (Constanţa County, Romania). Our goal was to define the physiographical features: structure, texture and firing parameters for this group of ceramic objects. The original 20 archaeological objects are represented by oil lamps (4), plates (8), bowls (6), one jug, and one terracotta – all in the form of fragments. From the technical realisation point of view, 13 objects were pressed in moulds, while the rest (7) were formed on the potter's wheel. Excepting three objects that lack decoration, the rest show various decorations: in relief, incised or printed.

The 10 ceramic fragments we have investigated are representative for the original group of 20 samples. They were investigated macroscopically, microscopically, by X-ray diffraction (XRD), and for their compaction characteristics: water adsorption, density and apparent density.

Based on their macroscopic and physical features, as well as on the mineral composition by XRD, we could separate two categories of ceramics fired at different temperature ranges: (1) 800°-900°C (6 samples), and (2) 900°-950°C (4 samples).

***Keywords**: Roman ceramics, mineralogical and physical analysis, Histria Romania*

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INTRODUCTION

Ceramic production started in prehistory. The discovery that firing modelled clay turns into ceramics represented a turning point in the development of humankind. When one studies ceramics for its historical signification, understanding the fabrication technology, the firing parameters and, in a limited measure, the raw materials as provenance area are important. They can be deduced based on the ceramics structure, texture, or mineralogical composition [1, 2, 3, 4].

Histria was a Greek colony founded in Dobrogea, near the Black Sea coast (nowadays on the border of Sinoe Lake) towards the middle of the 7th century B.C.. It lasted for 13 centuries, till the 7th century A.D.. Histria is the oldest Greek colony on the western shore of the Pontus Euxinus and one of the first ones build around this sea. At the same time, it represents the first town officially attested in the present-day territory of Romania [5, 6]. Archaeological investigations in the Greek-Roman fortified town started in 1914, coordinated by Prof. V. Pârvan. Ever since, generations of researchers, historians and archaeologists have dedicated their efforts in studying the Milesian settlement. This makes Histria one of the most well-studied and known colonies around the Pontus Euxinus. Currently, 15 monographic volumes are published; besides, numerous studies illustrate various archaeological finds, as well as a wide insight into the political, economic, social and cultural life of this key Greek-Roman town (see series *Histria*[1], volumes I-XV, of which *Histria V* is dedicated to the ceramic workshops).

The paper presents the result of the mineralogical and physical investigation on 10 Roman ceramic fragments (oil lamps, bowls and plates) carefully selected from the total of 20 samples collected from the west-east section, *Basilica extra muros* Sector from Histria. The ceramic objects illustrate a wide range of usages, from luxury objects to the daily use ones, including lightning purposes. They were unearthed during the 2002-2004 archaeological campaign (for the archaeological overall results, see [6, 7, 8, 9]).

Our goal was to establish the ceramics physiographical characteristics: structure, texture and firing parameters. We also included a brief presentation of the geology of Central and South Dobrogea, as background information on the potential local sources of clay raw materials. All the original 20 samples have been macroscopically characterized; based on these features we have selected the 10 fragments for further investigation.

[1] http://www.cimec.ro/Arheologie/web-histria/6bibliografie/bibliografie_eng.htm

RESULTS AND DISCUSSION

Macroscopic characterisation

The categories of objects identified in the archaeological site are oil lamps (4), plates (8), bowls (6), one jug and one terracotta figurine; all are present as fragments. From the technical realisation point of view, 13 objects were pressed in moulds, while the rest (7) were formed on the potter's wheel. Excepting three objects that lack decoration, the rest show various decorations: in relief, incised, printed or just coloured stripes on the outer side.

In general, the ceramic paste has a homogeneous colour varying from various red hues to reddish brown [10], suggesting oxidizing firing regime. Only samples 13 and 14 (Figs. 1 and 3) show areas with distinctive colorations: within the light red paste, there are different coloured layers towards the inner, and respectively the outer surfaces. The matrix is also relatively homogenous, while no clast aggregates were noticed. The matrix (structural/grain-size) fineness varies from fine to semifine. The clasts are angular to rounded and show various sizes (up to 2 mm). Macroscopically, we noticed quartz grain and muscovite lamellae as mineral components. Porosity varies in limited range; both mainly elongated primary pores lined parallel to the surfaces, and secondary pores with circular or irregular outlines generate it. The pore sizes range between 0.5 x 1.5 and 1.5 x 2.0 mm.

The archaeological information and the macroscopic characteristics of the samples selected for further study among the original set are presented in Tables 1 and 2.

Table 1. Archaeological information on the studied ceramic fragments

Sample no.	Inventory no. / Archaeological source	Object type / Observations
1	52/2004 S I, square 4, -2.20-2.30 m depth, infilling of oven no. 5	Oil lamp; imported product
2	35/2003 S I, square 8, -1.10 m depth, southern profile, at the level of the phase II B floor	Oil lamp; local product (?)
4	31/2004 S I, square 1, -1.70 m depth, in the area of oven no. 1	Oil lamp; imported product
5	6/2003 S I, square 3, -1.64 m depth, underneath the wall foundation in room A (phase II B)	*terra sigilata* plate; imported product
11	9/2003 S I, square 5, -0.73 m depth, in the filling layer between phases IV A and II B	Bowl Drag. 24; *Eastern sigillata* C; imported product
12	53/2003 S I, square 9, -1.45 m depth, from the demolition layer of the room with mortar-bound wall and wood wall, phase I C	Plate; local product

Sample no.	Inventory no. / Archaeological source	Object type / Observations
13	14/2002 S I, square 5, -1.60-1.70 m depth, at the level of the smith workshop	Bowl; local product; traces of secondary firing on the outer surface
14	40/2003 S I, square 7-8, -1.46 m depth, from the yellow loam floor, phase II A	Bowl; local product, possibly a refuse; *graffiti* in Greek on the bowl's body surface
16	79/2004 S I, square 5, -2.60 m depth, from the infilling of oven no. 8	Bowl; local product (?)
20	8/2003 S I, square 4, -1.50-1.70 m depth, from the ditch underneath the organisational level of phase IV A	*terra sigillata* plate; local product (?)

Table 2. Macroscopic characteristics of the studied ceramic fragments

Sample no.	Paste (quality, Munsell colour, components)	Engobe	Decoration
1	- fine, reddish-brown (2,5YR, 7/8), with iron oxides and fine muscovite lamellae	Red shiny engobe, with golden metallic lustre at the outside	The lid shows a relief decoration with vine leaf and tendril
2	- fine, light red (10R, 7/8), with fine fragments of quartz and muscovite lamellae	Red engobe at the outside	The lid is shell-like with relief leaves on the lateral side
4	- fine, light red (10R, 6/8), with fine fragments of quartz and muscovite lamellae	Red shiny engobe of extremely high quality at the outside	Half-oval printed decoration on the side, incised decoration in the central area of the lid
5	- fine, reddish brown (2,5YR, 7/6), with fragments of quartz and muscovite	Red shiny engobe with metallic lustre, of extremely high quality	Incised decoration on the side, in relief on the handle (of vegetal inspiration?); three flutings in relief marking the transition from the plate's rim to the body
11	- fine, light red (10R, 4/8); with fine muscovite lamellae	Dark red engobe of extremely high quality	Incised decoration
12	- fine, light red (10R, 4/8), with fine muscovite lamellae	Brown reddish engobe on ¾ of the outer surface; inside, only on the rim	Not decorated
13	- semifine, light red (10R, 4/8), with grey stripe in the middle (10R, 6/1, reddish grey) as a	-	Decoration represented by flutes in the upper part of the body

Sample no.	Paste (quality, Munsell colour, components)	Engobe	Decoration
	result of incomplete firing; quartz grains of variable sizes		
14	- semifine, light red (10R, 6/8), with lighter stripe at the exterior (7.5YR, 8/4, pink); with quartz fragments	Various hues of brown reddish engobe at the outer surface	Not decorated
16	- fine, reddish brown (2,5YR, 4/4), with quartz and muscovite fragments	Dark brown engobe, shiny on both sides	Fine flutes below the rim and above the maximum diameter
20	- fine, reddish brown (2,5YR, 7/8), with fine fragments of quartz	Engobe of the same colour as the paste	Decoration in relief: a. hunting scene; b. animals (goats) in motion; c. male mask, left side profile, with frontal caduceus

Figure 1. Sample 14 - bowl

Figure 2. Sample 20a – plate

Figure 3. Sample 13 - bowl

Figure 4. Sample 2 - oil lamp

Figure 5. Sample 1 - oil lamp

Figure 6. Sample 12 – plate

Optical microscopy

We have used transmitted light optical microscopy for completing the information on the mineral components of the matrix and the flux, for identifying the thermal transformations thus defining the firing temperature, and for assessing the fineness, *i.e.*, the fabric of the studied ceramics.

The matrix consists of thermally affected clay minerals and, to a lesser extent, of quartz, feldspars and micas. In parallel polarized light, one nicol, it is light brown-yellowish gradually passing into reddish brown, dark brown or even black (sample 13 – Figure 9) as a function of increasing firing temperatures. Similarly, under crossed nicols, the matrix displays birefringence with intensities decreasing with increase of firing temperature. In addition, we noticed transition areas from the inner to the outer surface of the ceramic fragment marked by darker coloration (sample 12 - Figure 12).

Grain size measurements on thin sections allowed us to separate two categories of ceramics: semifine (lutitic-siltic-arenitic type) and fine (lutitic-siltic type) (lutite < 0.004 mm, silt = 0.004-0.063 mm; arenite = 0.063-2 mm) [11, 12, 13]. Based on the ratio of crystalline components vs. amorphous phases in the ceramic matrix, we separated three fabric types: microcrystalline (samples 2, 4, 5, 14, and 20), microcrystalline-amorphous (samples 1, 11, 12, and 16) and amorphous-microcrystalline to amorphous, in only one sample (13).

The primary elongated pores are dominant (2–3 %, in samples 1, and 13) and do not exceed 0.55 x 1.50 mm. Reinforcing the preferential orientation of the mica lamellae, the orientation of the primary pores ensures a flow texture that is obvious in most of the ceramic samples.

The crystalloclasts reflect the mineralogical and petrographical diversity of the raw and the flux materials. Quartz crystalloclasts are ubiquitous (up to 3–5 %, maximum sizes of 0.35 x 0.70 mm); they are subangular to rounded, and sometimes display undulatory extinction and a variable degree of inner fissuring due to firing. Twinned plagioclase feldspars are present in subordinate amounts; they are sometimes sericitized and kaolinitized, with maximum sizes of 0.10 x 0.15 mm. Fine mica (muscovite and rarer biotite) lamellae, as a rule with relatively high birefringence pointing to firing temperatures below 800 - 850°C, were also noticed. Besides, iron oxi-hydroxides aggregates (mainly hematite grains of up to 0.03 x 0.08 mm), spinel fragments (up to 0.1 x 0.15 mm) and in sample 13 small zircon grains (0.08 x 0.10 mm) are present. The identification of calcite films (up to 0.17 x 0.30 mm in size) in sample 12 suggests relatively low firing temperatures (~ 800°C).

Among the lithoclasts, only quartzite fragments were identified, with maximum sizes of 0.80 x 1.05 mm representing not more than 2 % of the material (samples 13 and 14). In some cases, we could notice undulatory extinction of quartz grains.

Ceramoclasts vary from zero (or sizes below the microscopic range) in samples 1, 5, 11, 13, and 16 to up to 3 % of the mass in the rest of them, with maximum sizes of 0.87 x 1.87 mm (sample 14).

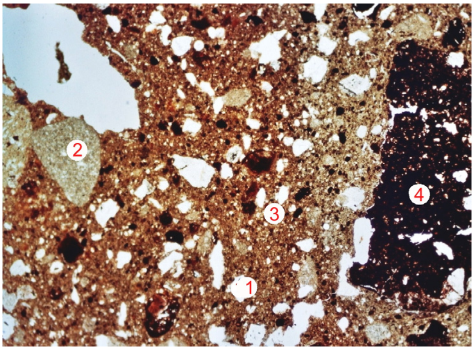

Figure 7. Sample 14 – semifine ceramics with microcrystalline matrix, with large pores; 1 quartz, 2 quartzite, 3 micas, 4 ceramoclast, 5 feldspar; 1N, 35x

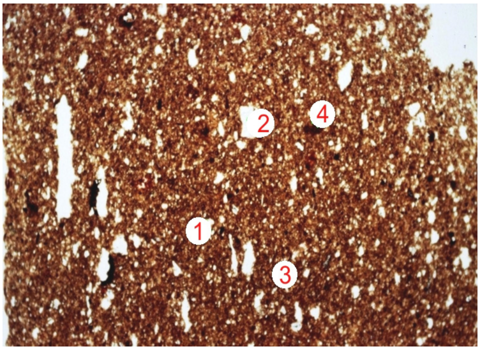

Figure 8. Sample 20 – fine ceramics with microcrystalline matrix, and elongated pores oriented parallel to the object's surface; 1 quartz, 2 quartzite, 3 micas, 4 ceramoclast; 1N, 35x

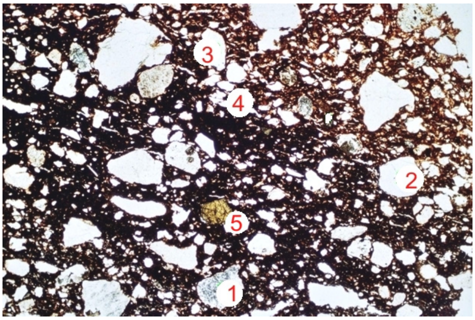

Figure 9. Sample 13 – semifine ceramics with amorphous-microcrystallin to amorphous matrix, and elongated pores; 1 quartzite, 2 quartz, 3 feldspar, sericitized and kaolinitized, 4 micas, 5 spinel; 1N, 35x

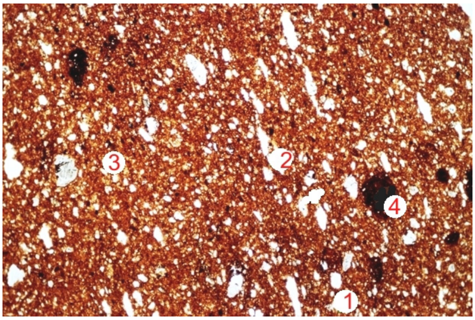

Figure 10. Sample 2 – fine ceramics with microcrystalline matrix, and elongated pores; 1 quartz, 2 quartzite, 3 micas, 4 ceramoclast; 1N, 35x

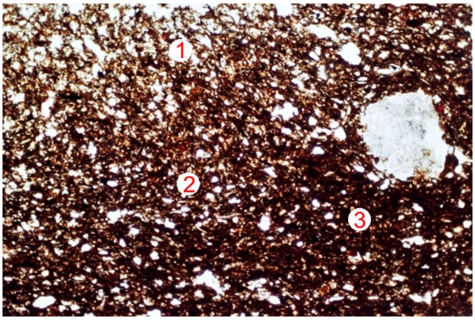

Figure 11. Sample 1 – fine ceramics with microcrystalline-amorphous matrix, and elongated pores oriented parallel to the object's surface; 1 quartz, 2 micas, 3 iron oxi-hydroxides aggregates; 1N, 35x

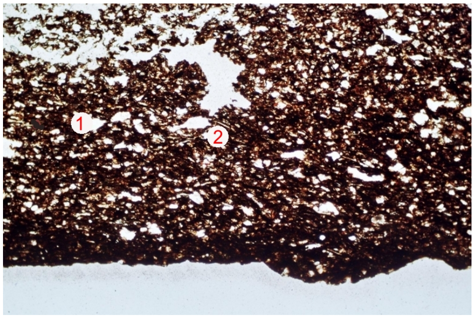

Figure 12. Sample 12 – fine ceramics with microcrystalline-amorphous matrix, and numerous elongated pores oriented parallel to the object's surface; 1 quartz, 2 micas; 1N, 35x

X-ray diffraction

The XRD patterns evidence a relatively simple mineralogical composition (Figs. 13 and 14). The main minerals, identified in all the studied samples, are quartz and Ca-Na feldspars (albite–anorthite). Clay minerals have been

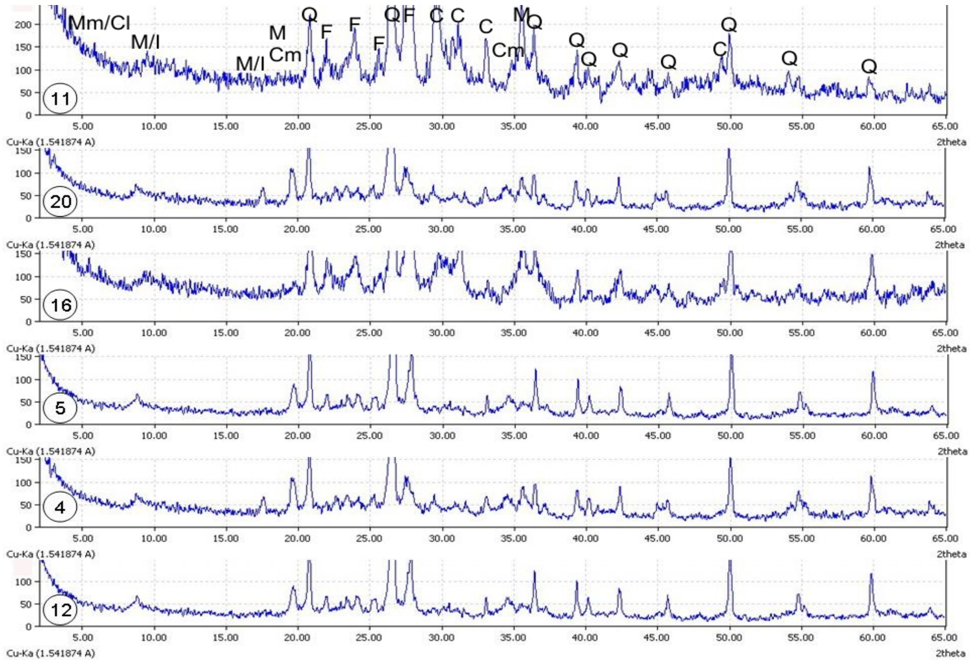

Figure 13. XRD patterns of the low firing temperature group of studied ceramic fragments; Q – quartz, F – feldspars (Ca-Na), C – carbonates (calcite), M/I – muscovite/illite, M – micas, Mm/Cl – montmorillonite/chlorite, Cm – clay minerals; firing temperatures between 800–900ºC; sample no. is indicated in the lower left corner.

thermally affected; they are illustrated only by the diffraction lines at 4.5 Å and 2.6 Å. Exceptionally, samples 1, 2, 13 and 14 lack the lines corresponding to micas and clay minerals, but show the XRD contribution of iron oxi-hydroxides (hematite/goethite). Carbonates (calcite) and micas (muscovite, muscovite / illite) have been identified in samples 4, 5, 11, 12, 16 and 20. We have assigned the 14 Å lines in sample 16 to montmorillonite–chlorite mixed-layers.

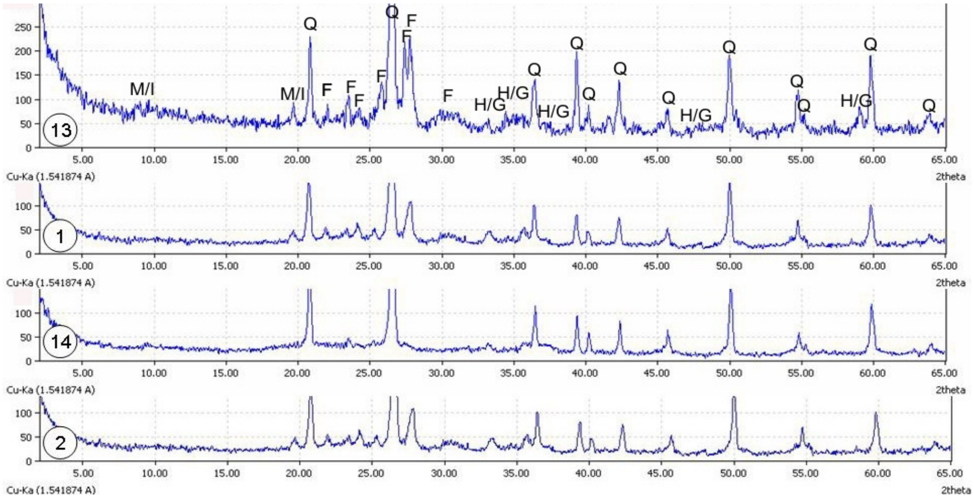

Figure 14. XRD patterns of the high firing temperature group of studied ceramic fragments; Q – quartz, F – feldspars (Ca-Na), H/G – hematite/goethite, M/I – muscovite/illite; firing temperatures between 900–950ºC; sample no. is indicated in the lower left corner.

Physical characteristics

From every ceramic sample under study, we have collected three fragments for physical characterization. The fragments were preserved in vacuum for 30 min, then dried in an oven and weighted. Then, they were placed in water for 2 hours and weighted afterwards again.

Table 3 summarizes the compaction characteristics of the ceramic samples, i.e., water adsorption, apparent density and porosity. The values vary to a little extent in the studied material, except for sample 1 that shows maximum values for water adsorption and porosity, and sample 2 that shows the minimum water adsorption value.

Table 3. Compaction characteristics of the studied ceramic samples

Sample no.	Water adsorption [%]	Apparent density [g/cm^3]	Apparent porosity [%]
1	22.944	1.605	36.825
2	9.959	2.019	20.104
4	18.363	1.700	31.217

Sample no.	Water adsorption [%]	Apparent density [g/cm^3]	Apparent porosity [%]
5	19.200	1.685	32.355
11	20.906	1.699	35.513
12	10.201	2.029	20.688
13	10.827	1.907	20.647
14	14.358	1.901	27.289
16	18.005	1.769	31.842
20	18.504	1.736	32.121

Based on the range of data obtained, we could group the samples into three groups:

Samples 4, 5, 11, 16, and 20 – with water adsorption values between 18.005 % and 20.906 %

Samples 2, 12, 13, and 14 (water adsorption between 9.959 % and 14.358 %)

Sample 1 (water absorption is 22.944 %).

The explanation for these distinctive behaviours resides in the macroscopic and microscopic features. Thus, sample 1 showing the higher water adsorption has a heterogeneous colouring pattern with darker colour towards the outer surface of the ceramic body, and numerous pores oriented parallel to it.

CONCLUSIONS

Among the 20 studied archaeological objects, there were 4 oil lamps, 8 plates, 6 bowls, one jug and a terracotta figurine, all present as fragments. 13 objects were pressed into moulds, while 7 were formed on the potter's wheel. Except for three non-decorated objects, the rest show various types of decorations (in relief, incised, printed or with stripes of various colours on the outer surface).

Macroscopically, the ceramic fragments are homogenously coloured, slightly varying from red to reddish brown, involving oxidizing firing regimes. Only samples 13 and 14 show distinctively coloured areas, as light red paste with layers of different colours towards both inner and outer surfaces.

The fineness (by structure/grain size) of the matrix varies from fine to semifine. The clasts are angular to rounded or irregular; their maximum sizes are below 2 mm. At macroscopic scale, we could identify only quartz grains and muscovite lamella as mineral components.

The microscopic investigation evidenced the presence of a matrix consisting of thermally affected clay minerals and crystalloclasts: quartz, plagioclase feldspars, micas, magnetite, hematite, calcite, zircon, and spinel. Quartz crystals are mainly angular; they sometimes show undulatory extinction. Feldspars are often sericitized, kaolinitized and polysynthetically twinned.

Carbonate films noticed in thin sections were also confirmed by the XRD patterns of samples 4, 5, 11, 12, 16, and 20. Lithoclasts are exclusively represented by quartzites, while ceramoclasts are present in almost all the samples. The grain size measurements performed under the microscope allowed the separation of two categories within our ceramic samples: fine (lutitic-siltic) and semifine (lutitic-siltic-arenitic). Based on the ratio between the crystalline components vs. the amorphous phases, we have separated three fabric types: microcrystalline, microcrystalline-amorphous and, in only one sample (13), amorphous-microcrystalline to amorphous.

A much darker matrix in some areas of the ceramic body reflects relatively higher local temperatures in the oven, leading to the transformation of the iron oxi-hydroxides.

Porosity varies to limited extent, between 1 to 5 %. It is represented by mainly elongated primary pores oriented parallel to the surface of the ceramic body, and by rounded or irregular secondary pores. Compaction, as evidenced by water adsorption values, ranges from 9.59 % and 27.65 %.

The mineralogical transformations at microscopic scale, such as fissures forming in the quartz grains, presence of iron oxi-hydroxides, matrix isotropization, presence of secondary pores, variable amounts of micas, and presence/absence of carbonates have allowed us to estimate the firing temperatures for the studied ceramics.

Thus, based on the microscopic observations and the XRD mineralogical composition, we could group our samples into two categories with distinctive firing temperatures: (1) 800°-900°C for samples 4, 5, 11, 12, 16, and 20; (2) 900°-950°C for samples 1, 2, 13, and 14.

Concerning the local raw materials, clays are frequent in Dobrogea. The Central Dobrogean Massif is a distinctive compartment tectonically lifted between the Capidavia-Ovidiu and Peceneaga-Camena faults. Its basement consists of a slightly metamorphosed sedimentary series, known as „the green schists series", and a mezometamorphic crystalline schists series [14, 15]. Locally, a slightly folded unconformable sedimentary cover consisting of Jurassic, Cretaceous and Tertiary epicontinental deposits (limestones, dolomites, conglomerates, sandstones, glauconitic sands, clays, and marls) overlies the basement. Clay occurrences possible use in ceramic industry outcrop in the neighbourhoods of Histria, Saele, Cogealac, and Sinoe localities.

In Central and South Dobrogea, thick deposits of loam of red-brownish clay formed on the top of the pre-Quaternary deposits. In the base of this succession, red and red-yellowish clays are known, frequently containing calcareous and Fe-Mn concretions, gypsum crystals and fragments of pre-Dobrogea rocks [16]. Complex studies on obtaining industrial ceramics were performed also by using the clays from Cobadin quarry (Constanţa County) [17].

EXPERIMENTAL SECTION

- the macroscopic investigation was performed by using a Nikon SMZ 645 binocular.
- the XRD patterns were obtained with a Shimadzu XRD6000 (Bragg-Brentano geometry) diffractometer, with Cu anticathode (Cu-K_a, λ_{Cu} = 1.541874 Å), 40kV, 30 mA, in the 2°–65° 2Theta interval, $\Delta 2\theta = 0.02°$.
- the microscopic study was performed on thin sections (< 25 μm) in polarized light by using a Nikon Eclipse E200 microscope. The microphotographs were taken with a NIKON FDX-35 camera.
- the physical characteristics (apparent density, water adsorption, apparent porosity) were determined based on the Law of Archimedes, by saturation in water and boiling of the ceramic fragments.

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