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**COMPOSITION AND TECHNOLOGY OF LOWER DANUBE KAOLIN WARE (LDKW)****Examples from *Novae*, Bulgaria**With a contribution by Silviu Rădan<sup>4</sup>**Introduction**

This paper deals with the results of laboratory analysis carried out on 13 pottery samples from the Roman *castrum* of *Novae*, situated on the right bank of the River Danube, formerly *Moesia Inferior*, currently Northern Bulgaria. Three samples from the aforementioned 13 fragments were analysed (DASZKIEWICZ ET AL. 2006) as part of a project carried out for the purposes of E. Klenina's doctoral thesis (KLENINA 2006). This pottery assemblage (totalling 72 sherds) was recovered from excavations conducted at *Novae* by the archaeological expedition team from Poznań's Adam Mickiewicz University, led by A. B. Biernacki. The other ten pottery sherds discussed in this report came from excavations carried out by the Archaeological Research Centre of the University of Warsaw, under the direction of Prof. P. Dyczek. They were selected and submitted for analysis by Prof. P. Dyczek. Four of the sherds underwent laboratory analysis as part of M. Baranowski's undergraduate thesis written at the Institute of Archaeology of the University of Warsaw (BARANOWSKI ET AL. 2006; BARANOWSKI/DASZKIEWICZ 2009), whilst analysis of the remaining six fragments was carried out as part of P. Dyczek's study of whitish-firing pottery. This pottery has recently been named Lower Danube Kaolin Ware (LDKW)<sup>1</sup>.

This paper concentrates exclusively on presenting the results of laboratory analysis. The already published analysis results for seven of the pottery fragments, as well as the previously unpublished analysis results for the remaining six fragments have been classified jointly herein, in uniform fashion adhering to the principles of classification set out below. The groups for LDKW pottery which were determined using this method can be used as a starting point in the future for provenance analysis.

According to E. J. Klenina,<sup>2</sup> vessels of the LDKW type found at *Novae* were made at workshops in *Singidunum* (KLENINA 2006, 114; 115; 178). This variety of pottery was widespread in the territories of *Moesia Superior* and *Inferior* and in Dobrudja (KLENINA 2006, 184). Pottery of this type found in the territories of Dobrudja is described, for example, by Radulescu (RADULESCU 1975). In P. Dyczek's view, archaeological evidence (the largest concentrations of LDKW finds) suggests that LDKW pottery was, or may have been, made at production centres in the Dobrudja region (Dyczek personal communication).

It is not possible to answer the questions posed by archaeologists (E. J. Klenina and P. Dyczek) relating to the geographical location of the production of the LDKW pottery found at *Novae* without analysing locally occurring kaolin raw materials<sup>3</sup> or kiln-site pottery of the LDKW type.

Unfortunately, thus far the authors of this article have been unable to analyse kaolinitic clays or other clay raw materials from Dobrudja, nor have they been successful in finding any analysis results in the literature which could be used for comparative purposes. Published results of chemical analysis carried out on kaolinitic clays from Dobrudja that only take into account the major elements (COMŞA 1985) can be taken into consideration in provenance studies only to a limited degree, as comparing major elements alone can lead to erroneous conclusions. Chemical analysis was not carried out on even a single LDKW sherd from the kiln site at Dobrudja, therefore the authors of this article have no chemical analysis results to refer to. In consequence, laboratory analysis has yet to provide any evidence for the LDKW ceramics discovered at *Novae* having been made at production centres in Dobrudja. Moreover, no sample of kiln pottery from *Singidunum* has been analysed, nor have any kaolin deposit samples from Serbia, hence there is also no evidence to suggest that the analysed sherds were not made at the *Singidunum* production centre. Bearing in mind the availability of kaolinitic clays none of these theories can be precluded. Furthermore, ceramic workshops did exist in *Novae* (either within or beyond the city walls, depending on the period) and produced both tablewares and ceramic building materials (KLENINA 2006<sup>4</sup>). As local production was insufficient to meet the city's needs, pottery vessels were imported from neighbouring production centres, such as Butovo,

<sup>1</sup> This name was introduced by P. Dyczek and does not appear in earlier articles. The term "Kaolin" was taken from the assumption that white firing clay with alumina contents above 20% should mainly consist of kaolinite. This should be proved with future studies of the raw clays.

<sup>2</sup> Sample MD2518 = pot TYPE 27 equating to a type of vessel produced at Singidunum from the late 2nd to the early 5th century (KLENINA 2006, 115; 164; 178); sample MD2542 = pot TYPE 25 equating to a type of vessel produced at Singidunum from the 3rd to the early 5th century (ibid. 114; 164; 178).

<sup>3</sup> As kaolin raw materials are regarded sediments with minimum 15% contents of the clay mineral kaolinite.

<sup>4</sup> Part 2 chapters 1–3, 157–158 (Polish summary), 71–172 (English summary).

Pavlikeni, Hotnica, and *Nikopolis ad Istrum*; this is evinced by both typological studies and laboratory analysis (KLENINA 2006; DASZKIEWICZ ET AL. 2006; DASZKIEWICZ/SCHNEIDER 2007). At the same time, pottery produced at *Novae* was also an export commodity, e.g. vessels discovered at *Iatrus* were made at two different workshops in *Novae* (DASZKIEWICZ/SCHNEIDER 2007). The existence of a local ceramic industry and the fact that kaolinitic clay deposits were accessible in the neighbouring territories (Senovo, Vetovo)<sup>5</sup> raises the question of whether some of the LDKW pottery found at *Novae* might have been produced at local ceramic workshops as imitations of vessels from Dobrudja. Unfortunately, this question must also remain unanswered for the time being; the only available comparative material is chemical analysis of major elements carried out on a single sample of kaolinite from Bulgaria (BENEA/GOREA 2004). Consequently, there was no answer based on laboratory analysis results to the question of where the 13 LDKW vessel fragments found at *Novae* were made: do they represent imports, regional production, or both? The only reference which could be quoted with regard to determining the provenance of these vessels was either E. J. Klenina's or P. Dyczek's hypothesis about imports.

In view of this situation, the authors turned to geologists from Romania to find a contact specialising in kaolin raw material deposits from Dobrudja<sup>6</sup>. This resulted in undertaking collaborative work with Silviu Rădan of the National Institute of Marine Geology and Geoecology - GeoEcoMar, who, for the purposes of this article, was kind enough to provide access to unpublished results from analyses carried out as part of his doctoral thesis. A comparison of results from the analysis of kaolin raw materials from Dobrudja carried out by S. Rădan with results obtained from the analysis of 13 LDKW vessels discovered in *Novae* reveals that only one vessel may have been made at a pottery workshop in Dobrudja. Twelve of the analysed LDKW vessels were made from raw materials of different chemical composition to that noted in the kaolin raw materials from Dobrudja examined by S. Rădan; it also differs from the chemical composition of kaolinitic clays given by M. Comşa (COMŞA 1985).

### *Kaolinitic clays of Dobrudja (by S. Rădan)*

The kaolinitic clays of South Dobrudja occur as more or less horizontal beds and lenticular beds, with variable thickness (up to 20 m) and variable lateral extent (hundreds of metres), associated with cross-bedded sands, gravels and very fine blackish and grey clays, rich in carbonaceous remains, which may include, in some places, thin lignite beds. The clays outcrop over a large area, between the localities of Hârşova, Cernavodă, Medgidia and Constanţa. These deposits represent the continental facies of the Lower Cretaceous (Aptian), represented by the so-called Gherghina Formation. The continental deposits overlie Upper Tithonic-Berriasian-Valanginian and Barremian limestones, or even older marine Aptian deposits, and are transgressively overlain by Upper Cretaceous, Sarmatian or Quaternary deposits.

The source area of these continental fluvio-lacustrine deposits is represented by the old weathering (lateritic) crust

developed in the North Dobrudja area, over granitic, dioritic or porphyric massifs and even on Palaeozoic sedimentary deposits, during a pre-Cenomanian stage (RĂDAN ET AL. 1972; RĂDAN 1989, 2000).

Clay mineralogy of the continental deposits is characterised by dominant kaolinite (70–90% of the under 2 microns fraction), followed by illite (10–30%) and smectite (5–20%), with accidentally vermiculite and chlorite. Among non-clay minerals, quartz (dominant), feldspars (rare), calcite (accidental), siderite and pyrite (only in carbonaceous clays) and goethite ± hematite (missing in the white clays, but frequent in the coloured ones) could be detected.

Kaolinitic clays are used in the manufacture of ceramic plates, building bricks, type moulds for foundries, for decoration and waterproofing, in the cosmetic industry and in dyestuffs. Mining activities take place at open quarries in three locations: Cuza Vodă, Țibrinu and Defcea, owned by the company S.C. Miniera Medgidia. At present, this company is one of the most important producers in Romania, extracting, processing and selling kaolinitic clay.

The kaolin deposits of Bulgaria are located over a karst relief of Lower Cretaceous limestones, and there is no agreement of opinion about the age and genesis (MANOLOV ET AL. 1968). The facies presented by these sedimentary kaolin deposits in Bulgaria are very similar to the Romanian deposits, but the stratigraphic context is different. The quality of the ceramic products should to be the same.

### **Laboratory procedures**

The following laboratory procedures were carried out: the matrix of each sample was examined using MGR-analysis (Matrix Group by Refiring), chemical composition of the sherd<sup>7</sup> was examined by WD-XRF, and the non-plastic components of the body were studied in thin-section under a polarising microscope (for a description of methods see Appendix). These three methods yield arguments for the provenance of vessels and for the technology used in pottery making. In addition, functional properties of individual pottery vessels were compared by gauging their water permeability and thermal shock resistance.

The first investigative procedure carried out<sup>8</sup> was MGR-analysis. All of the samples were refired at three temperatures: 1000°C, 1100°C and 1200°C. The thermal behaviour of the sample refired at these temperatures encompasses the

<sup>5</sup> Near Ruse; the distance between *Novae* and these kaolin clay deposits is c. 30 km as the crow flies.

<sup>6</sup> The authors would like to thank Dr. Antoneta Seghedi of the Geological Institute of Romania, National Museum of Geology Romania, who made contact with Dr. Silviu Rădan possible.

<sup>7</sup> Chemical analysis reveals the geochemical characteristics of both the plastic and non-plastic components of a pottery fabric. It enables the quantity of major and trace elements in the body to be established, although the phases in which individual elements occur cannot be ascertained (giving the major elements as oxides is standard procedure in geochemistry when presenting the results of chemical analysis).

<sup>8</sup> The term "carried out" refers to the previously unpublished analysis of six pottery fragments; in the case of the other seven samples earlier completed and already published analysis results were used.

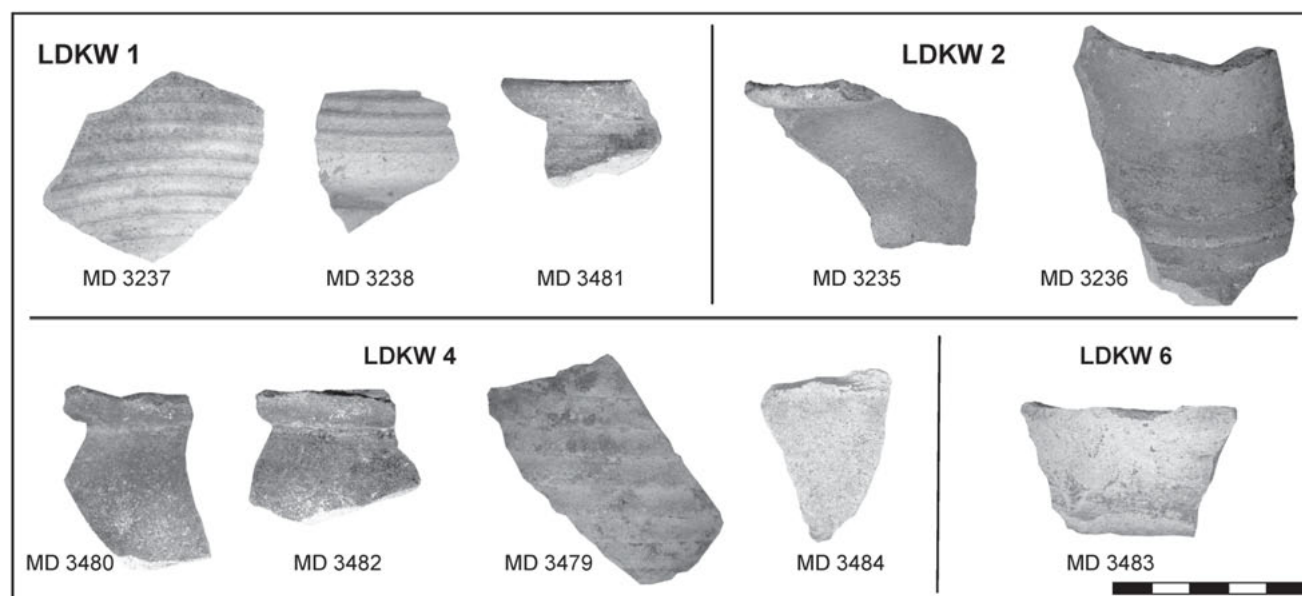


Fig. 1. Some of the analysed pottery fragments, outer surfaces.

Sample No.	Samples given by:	Manuscripts concerning these samples		Laboratory analysis				
		archeological	laboratory analysis	MGR	ts	XRF	FP	CP
MD 2518	A.B.Biernacki/	Klenina 2006	Daszkiewicz et al. 2006	8 temp.		+		+
MD 2535	E. Klenina			8 temp.		+	+	+
MD 2542				8 temp.	+	+	+	+
MD 3235	P. Dyczek	Baranowski's	Baranowski et al. 2006	8 temp.	+	+		+
MD 3236		undergraduate thesis	Daszkiewicz et al. 2007, unpublished	8 temp.		+		+
MD 3237		IA UW, 2007	Baranowski & Daszkiewicz 2009	8 temp.		+		+
MD 3238		(unpublished)		2 temp.		+		+
MD 3479	P. Dyczek	P. Dyczek poster Cádiz 2008	Daszkiewicz et al., this volume	3 temp.	+	+	+	+
MD 3480				3 temp.	+	+		
MD 3481				3 temp.	+	+		
MD 3482				3 temp.		+		
MD 3483				3 temp.	+	+	+	+
MD 3484				3 temp.		+		

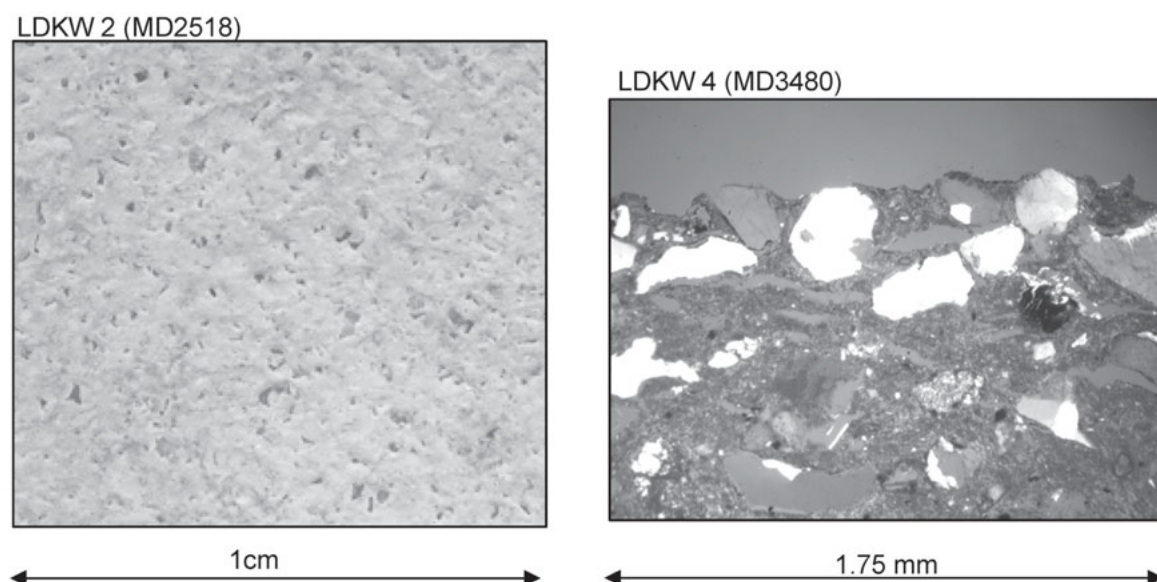
**Table 1.** List of analysed pottery fragments of LDKW found in *Novae*. MGR = MGR analysis; temp. = number of temperatures at which refiring was made; ts = thin-section; XRF = chemical analysis by WD-XRF; FP = functional properties (water absorption and thermal shock resistance); CP = ceramic properties (open porosity, water absorption, apparent density).

sample's appearance as well as its colour after refiring; both are taken into account when defining different matrix types (MGR groups). Definitive classification is based on thermal behaviour at 1200°C. If samples display the same thermal behaviour, colour and shade after refiring at 1200°C this indicates that they were made using the same plastic raw material<sup>9</sup>. This means that MGR groups are groups of greatest similarity pointing to a single workshop. All ceramic samples belonging to the same MGR group were made of the same clay, or of the same ceramic body.

The second stage of this study was chemical analysis which enabled the MGR-groups to be categorised according to the sherds' geochemical composition. This procedure allowed clay types, production areas, production centres, and workshops to be determined. Clay types (kaolinitic, illitic

etc.) equate to groups identified on the basis of bulk chemical analysis – these are groups of samples of similar contents of major elements (a precise assessment of the type of clay minerals is impossible because of the fact that they undergo thermal decomposition during the firing process). Production areas equate to samples characterised by similar geochemical parameters, whilst production centres (refer-

<sup>9</sup> Experience gained from carrying out MGR-analysis on eight thousand samples of clay and pottery from numerous sites of various date from Europe, the Near East, Egypt, Sudan and the New World clearly shows that terminating refiring at a lower temperature may lead to erroneous conclusions because, in many instances, after refiring at lower temperatures, even originally very low fired samples may look very similar (or identical), but will differ significantly after refiring at higher temperatures (DASZKIEWICZ/SCHNEIDER 2001).



**Fig. 2.** Unsmoothed surfaces of LDKW are due to the presence of non-plastic inclusions of the grains of quartz and feldspars in medium sand fractions. Left: outer surface of LDKW 3 (MD2518), macrophotograph by M.Baranowski. Right: photomicrograph (XPL), vertical to outer surface of LDKW 4.

ence groups) equate to sub-groups determined within one production area. Workshops equate to groups of samples distinguished within particular production centres; they are groups of greatest similarity, i.e. samples of the same chemical and phase composition which were made at the same workshop (not infrequently from the same production batch).

One clay type may have been used at several production areas and production centres, whilst a number of workshops may have operated within each production centre. It has to be borne in mind that the term workshop (as well as term production centre) can be interpreted both in their territorial and chronological sense. Various raw materials may have been in use at several contemporaneous workshops (or even a single workshop), as well as at workshops dating from various periods (correlation raw material – dating).

The next step of analysis involved thin-section studies, which allowed for verification of whether the ceramic body used at specific workshops/production centres featured the same type of non-plastic components (natural inclusions or intentional temper)..

In addition, MGR-analysis was carried out on six samples at temperatures of 400°C, 600°C, 700°C, 800°C and 900°C in order to determine their equivalent original firing temperature. The functional properties of only four sherds were assessed in view of the small size of the samples. The ceramic properties (open porosity, water absorption and apparent density) of nine of the pottery fragments were measured. An evaluation was made of whether there was any correlation between macroscopically observed similarities in fabric and the groups determined by laboratory analysis. A list of the samples analysed is presented in **table 1**, some of the analysed samples, divided into provenance groups determined by laboratory analysis are shown in **figure 1**.

## Results of classification

All analysed LDKW pottery sherds are characterised by similarly coloured outer and inner surfaces, and by the encircling groove decoration noted on many of them. Another characteristic feature of these samples is the appearance of their surfaces (**fig. 2**); these are unsmoothed and quite coarse due to the presence of non-plastic inclusions in medium sand fraction (**fig. 3**). Patches of a thin brown-red layer suggestive of slip, which wore off with use, are visible on the surface of two ceramic sherds (MD2518 and MD2535) in several places (DASZKIEWICZ ET AL. 2006). They most probably represent vestiges of painted decoration rather than the remains of slip which would have coated the vessel's entire surface.

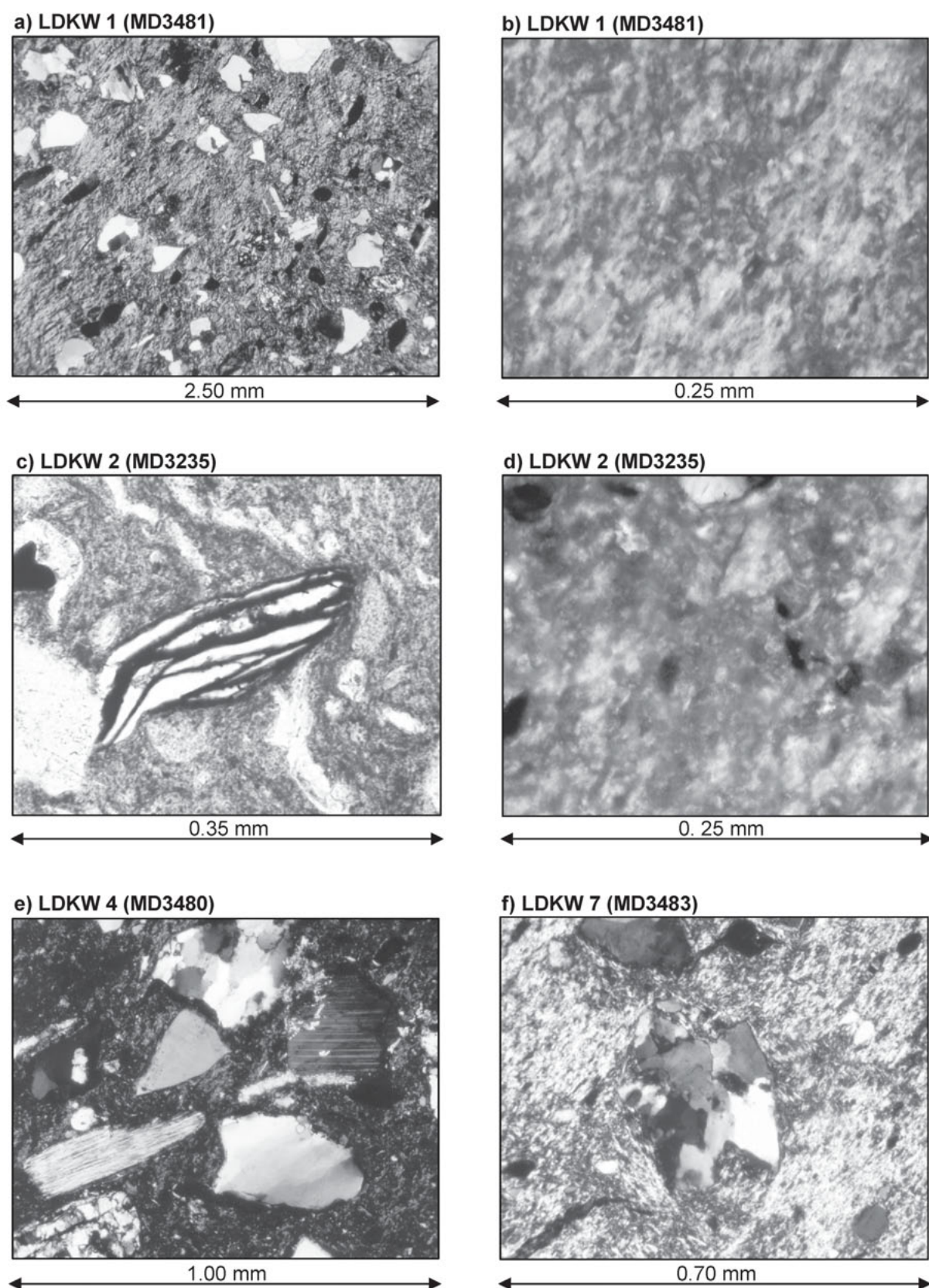
MGR-analysis led to two groups being determined based on the samples' appearance after refiring at 1200°C. Three of the samples have a sintered matrix type<sup>10</sup>, whilst ten samples have an over-fired matrix type<sup>11</sup>. Based on the colour of matrix after refiring at 1200°C only one category of matrix can be identified: a non-calcareous iron-pure matrix. Different colours and shades can be distinguished within this category of matrix; at 1200°C samples fire to various shades of beige or yellow. The results of MGR-analysis led to the conclusion that the 13 analysed samples represent nine MGR-groups<sup>12</sup> (**table 2**).

<sup>10</sup> Sintered (SN) = the sherd is well compacted, it may or may not become smaller in size in comparison to the original sample, whilst its edges remain sharp.

<sup>11</sup> Over-fired (ovF) = the sample changes in shape, bloating, however, does not occur, nor does the surface of the sample become over-melted.

<sup>12</sup> The word 'group' is also used in reference to single samples, as it is highly probable that representation by only one sample is merely accidental.





**Fig. 3.** Photomicrographs of thin-sections, XPL. **a)** LDKW1 (sample MD3481): typical fabric in thin-section, mainly grains of angular quartz, plagioclases; **b)** LDKW 1 (sample MD3481) matrix without grains of silt fraction; **c)** LDKW 2 (sample MD3235): dark-brown organic residue or fibrous mineral; **d)** LDKW 2 (sample MD3235): only very few non-plastic components were noted in the fraction below 0.01 mm; **e)** LDKW 4 (MD3480): grains of quartz, polycrystalline quartz, plagioclase and muscovite; **f)** LDKW 7 (MD3483): grains of polycrystalline quartz.

Sample No.	MGR-analysis			MGR groups (works.)	Macroscopic characterisation			Ceramic properties		
	T <sub>eq</sub> [°C]	appear. at 1200°C	colour at 1200°C		Pores shape	Texture of fresh break		Po [%]	N [%]	d <sub>v</sub> [g/cm <sup>3</sup> ]
MC 3237	1000 - 1100	ovF	greyish-beige	1	elongated	paraller	spongy	21,6	10,9	1,97
MC 3238	-	ovF	greyish-beige	1	elongated	paraller	spongy	20,7	10,4	1,97
MC 3481	< 1000	ovF	greyish-beige	2	sligh.elongated	chaotic	dence with pores	-	-	-
MC 2518	900 - 1000	SN	yellowish-greyish	3	elongated	paraller / net	spongy	27,9	14,9	1,87
MC 3235	1000 - 1100	ovF	brownish-beige	4	sligh.elongated	net	spongy	22,9	11,7	1,96
MC 3236	1000 - 1100	ovF	brownish-beige	4,1	sligh.elongated	net	spongy	28,3	15,2	1,85
MC 2535	1000 - 1100	SN	orange-greyish	5	-	-	spongy	30,9	16,9	1,83
MC 3480	1000 - 1100	ovF	brown-beige	6	sligh.elongated	net	spongy	-	-	-
MC 3482	1000 - 1100	ovF	brown-beige	6	sligh.elongated	net	spongy	-	-	-
MC 3479	1000 - 1100	ovF	yellow-greyish	7	sligh.elongated	paraller / net	slightly spongy	24,9	12,8	1,95
MC 3484	1000 - 1100	ovF	yellow-greyish	7	sligh.elongated	paraller / net	slightly spongy	-	-	-
MC 2542	900 - 1000	SN	yellowish-greyish	8	sligh.elongated	paraller / net	spongy	25,7	13,1	1,95
MC 3483	< 1000	ovF	greenish-beige	9	various	chaotic	dense	24,2	13,1	1,84

**Table 2.** Results of MGR-analysis, macroscopic description of pores and texture of fresh break, and values of ceramic properties. T<sub>eq</sub> = equivalent original firing temperature; ovF = over fired; SN = sintered; sligh. = slightly; works. = workshops; Po = open porosity; N = water absorption; d<sub>v</sub> = apparent density.

The results of chemical analysis (**table 3a**) revealed large differences in the content of Al<sub>2</sub>O<sub>3</sub> (c. 19–27%) and discrepancies in the content of TiO<sub>2</sub> (0.67–1.03%). The level of SiO<sub>2</sub> (c. 65–72%) does not always correlate to the Al<sub>2</sub>O<sub>3</sub> content (where increased SiO<sub>2</sub> content is linked to the addition of temper in the form of quartz grains the Al<sub>2</sub>O<sub>3</sub> content decreases), hence it should be characteristic of clay raw material deposits. Multivariate cluster analysis<sup>13</sup> led to the identification of seven chemical groups (LDKW 1–LDKW 7). Group LDKW1 is very distinctive in having a high Al<sub>2</sub>O<sub>3</sub> content (c. 27%) and a low content of SiO<sub>2</sub> (65%). Samples attributed to groups LDKW 2 and LDKW 3 have a CaO content of less than 0.9%, distinguishing them from samples with a c. 2% CaO content belonging to groups LDKW 4–LDKW 6. Samples from group LDKW 4 differ only slightly in chemical composition (primarily in their TiO<sub>2</sub> content) from LDKW 5 samples. The solitary sample (MD3483) representing LDKW 7 differs from the remaining samples in having an approximately twofold lower content of Na<sub>2</sub>O, marginally higher contents of K<sub>2</sub>O and rubidium (a geochemical correlate of potassium), as well as a higher zirconium content.

The seven groups (production centres) derived from the results of chemical analysis are obvious in thin-section studies. The thin-section of LDKW1 (sample MD3481) shows grains of angular quartz, plagioclases, few grains of polycrystalline quartz, some kalifeldspars, some muscovite, some rock fragments (quartz +sericite). Two small pieces of hornblende are also visible. Very few grains of calcite are visible within individual pores. Non-plastic components are mainly in the 0.25–0.5 mm fraction (**fig. 3a**). Less temper is observable in this sample in comparison to samples from LDKW1–LDKW6 groups. No non-plastic components were noted in the fraction below 0.01 mm<sup>14</sup> (**fig. 3b**). The thin-

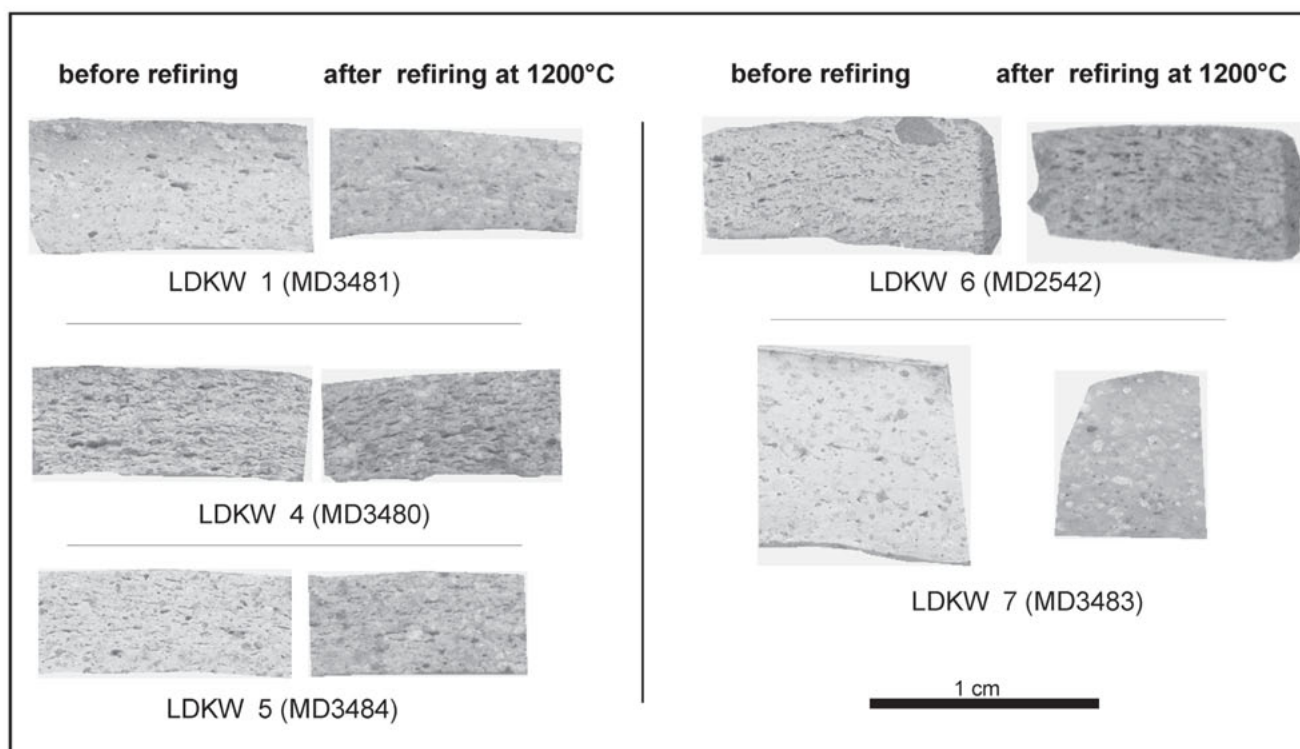
section of LDKW 2 (sample MD3235) shows a large number of quartz and feldspar grains in the 0.25–0.5mm fraction, as well as isolated grains of mica (0.7mm); some fragments of dark-brown residue from an unknown organic material or fibrous mineral (?) was also observed (**fig. 3c**). Only very few non-plastic components were noted in the fraction below 0.01 mm (**fig. 3d**). No thin-sections were made for LDKW 3 samples. The thin-section of LDKW 4 (MD3480) revealed grains of quartz (some grains of polycrystalline quartz), plagioclases and kalifeldspars (single piece of microcline) mainly in the 0.2–0.7 mm fraction (single grain of quartz up to 1.2 mm), a lot of calcite within the pores, some small pieces of chert, some muscovite (**fig. 3e**), single tiny piece of hornblende and some fragments of dark-brown (or opaque) residue from an unknown organic material or fibrous mineral (?), as did the thin-section of LDKW 5 (MD3479). LDKW 4 differs from LDKW 5 in having grains mainly in the 0.2–0.6 fraction and larger number of pores filled with calcite. Additionally the thin-section of LDKW 5 (MD3479) shows some grains of quartzite (one grain of quartzite up to 1 mm). Examination of thin-sections of LDKW 6 (sample MD2542) revealed the presence of plates of micas (0.04–0.33 mm), quartz grains and feldspars (up to 1.25 mm). Nearly all grains are in sand fraction<sup>15</sup>, only an insignificant proportion of non-plastic components in silt fraction were observed. Isolated iron-rich clay aggregates with

<sup>13</sup> Cluster analysis using Euclidean Distance and average link, elements: Si, Ti, Al, Fe, Mn, Mg, Ca, Na, K, V, Cr, Ni, Zn, Rb, Sr, Y, Zr and Ba (Brookhaven Data Handling Programs, kindly provided by E. Sayre).

<sup>14</sup> Particle sizes below 0.01 mm (10 µm) are defined as matrix.

<sup>15</sup> Classification of grain size fractions after Udden & Wentworth: clay <0.0039mm; silt 0.0039–0.0625mm; sand 0.0625–2.0mm; granules >2.0mm.





**Fig. 4.** Pottery fabrics: original sample (left) and sample after refiring at 1200°C (right), macrophoto of cross-cut.

quartz (up to 1.9 mm) are visible in thin-section (microphotographs of this thin-section can be seen in DASZKIEWICZ ET AL. 2006) as well as macroscopically (**fig. 4**). In the thin-section of LDKW 7 (MD3483) less temper is visible, only grains of quartz and polycrystalline quartz (0.15–0.5 mm) and two tiny pieces of hornblende were observed (**fig. 3f**).

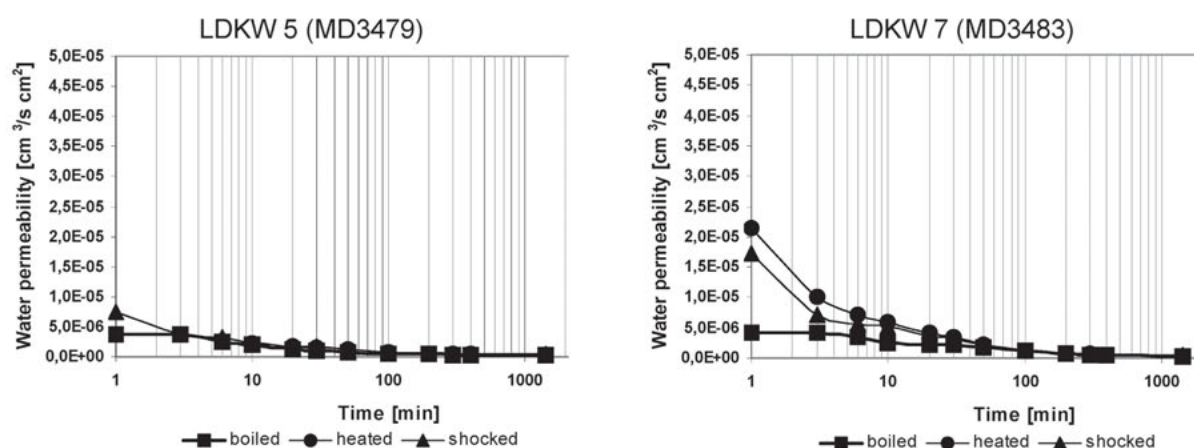
LDKW vessels were fired in an oxidising atmosphere. Only four samples were found to have an original firing temperature of below 1000°C, the remaining nine samples all having been originally fired at 1000–1100°C. The samples are very porous for such a high firing temperature. Their open porosity value ranges from 20 to 31% (**table 2**). The large number of pores is visible to the naked eye. These pores are linked to the vessel shaping process, and moreover, in virtually all samples they represent the result of the de-airing process having been carried out without due care. Only two fragments of pottery (one produced at workshop 2 and one fragment produced at workshop 9) were made from a more thoroughly de-aired ceramic body (random pores). The remaining samples can be divided into two groups based on their pore patterns: samples which have elongated pores that lie parallel to the surface of the vessel, and samples with lightly elongated pores forming a net-like pattern.

In terms of texture, the samples can be divided into those with a spongy (slightly spongy) texture and those with a texture dense with pores (**table 2, fig. 4**). These traits are visible both before and after refiring; up to 1200°C the samples remain poorly dense. The observation of a network of pores at this temperature is linked to the fact that the samples are made from a raw material which does not start melting until 1200°C (**fig. 4**).

Analysis of functional properties revealed that all four of the LDKW vessel fragments examined are impermeable and resistant to thermal shock. One of the vessels (LDKW 7) exhibited a change in permeability when a reading was taken after heating at 400°C (**fig. 5**). This indicates that the pores within these sherds contained organic matter blocking the flow of liquid – a result of the vessels' having been used. It may be linked to the lower firing temperature of sample LDKW 8; however, it does not have any significant effect on either vessel's functional properties. These vessels are very suitable for cooking and storing liquids – they would have made perfect cooking pots.

### Provenance

As already mentioned in the introduction, the results obtained by M. Comşa from analysis of kaolin raw materials (COMŞA 1985, 99) were available for comparative purposes, as were the as yet unpublished results of analysis carried out by S. Rădan. The chemical composition results for kaolinitic clays published by M. Comşa are, unfortunately, of little use for provenance studies in view of the fact that chemical composition is given only in the form of ranges, with the content range of particular elements being broad. Furthermore, analysis was carried out taking into account only seven major elements (Si, Ti, Al, Fe, Mg, Na, K), with K<sub>2</sub>O and Na<sub>2</sub>O contents being given additionally as a combined total (**table 3b**), whilst loss on ignition is given as a specific value (5.8%). However, these analysis results do not add up to 100%, even taking into account loss on igni-



**Fig. 5.** Water permeability versus time (logarithmic scale). Left: LDKW 5 (MD3479). Right: LDKW 7 (MD3483). Both vessels are impermeable and resistant to thermal shock. LDKW 7 exhibited a change in permeability after heating at 400°C, this indicates blocking the flow by organic matter accumulated in open pores.

tion and probable CaO contents. Thus, using this analysis in order to determine the precise provenance of LDKW is entirely futile, all the more so as in this instance Na<sub>2</sub>O and Zr contents are of particular significance (see the description of S. Rădan's analysis results given below), whilst M. Comşa gives Na<sub>2</sub>O content as a combined total with that of K<sub>2</sub>O, omitting the content of Zr altogether. Nonetheless, comparing results given by M. Comşa (calculated with the loss on ignition to fired clay) with those obtained from the chemical analysis carried out on 13 fragments of LDKW pottery recovered from *Novae* leads to the conclusion that this was not the raw material used for making the analysed ceramics (e.g. the contents of TiO<sub>2</sub> after calculation to fired clay is significantly higher).

S. Rădan's study of kaolinitic clays represents the results of analysis carried out on ten samples from Cuza Vodă, two samples from Gherghina, three samples from Mircea Vodă and one from Ovidiu (**tab. 3c**, data calculated to fired clay). These outcrops lie in southern Dobrudja, in an area bounded by ancient *Axiopolis – Histria – Tomis – Sacidava* (**fig. 6**). The content of major elements was determined for all clay samples, with the content of trace elements being determined for eleven samples. These analysis results reveal that kaolinitic clay deposits in southern Dobrudja are very varied in terms of their silicon dioxide content (59–76% SiO<sub>2</sub>), as well as their contents of Al<sub>2</sub>O<sub>3</sub> (15–33%), TiO<sub>2</sub> (0.75–1.49%), Fe<sub>2</sub>O<sub>3</sub> (1.18–4.91%), K<sub>2</sub>O (0.77–3.27%) and MgO (0.11–4.14%). Much smaller variations were observed in the content of CaO (0.74–2.22%) and Na<sub>2</sub>O (0.10–0.34%). A very characteristic feature of these kaolinitic clays is their relatively high content of iron compounds. All of the samples had low Cr and Ni contents. Zr content ranges from 135 to 367 ppm. Within individual outcrops the Zr/SiO<sub>2</sub> ratio is quite varied: for ten samples from Cuza Vodă it falls within a range of 2.09–4.54 (only single analyses are available for the remaining outcrops; the sample from Gherghina has a Zr/SiO<sub>2</sub> ratio of 5.99 and the sample from Ovidiu, 4.27).

The Zr/SiO<sub>2</sub> ratio in LDKW samples from *Novae* falls within a range of 1.63–2.06 for LDKW 1–LDKW 6, hence

it is lower than that of kaolinitic clays from Dobrudja. Only the sample representing the LDKW 7 group has a higher ratio which corresponds with the range of values for samples from Cuza Vodă (sample MD3483 has a ratio Zr/SiO<sub>2</sub> of 2.95).

A characteristic feature of the kaolinitic clays from Dobrudja is their very low content of Na<sub>2</sub>O (0.10–0.34%), whereas the Na<sub>2</sub>O content of ceramic samples representing LDKW 1–LDKW 6 is twice as high. Only the sample representing group LDKW 7 has an equally low Na<sub>2</sub>O content, and it is the same sample which has the same Zr/SiO<sub>2</sub> ratio as that noted in kaolinitic clays from Cuza Vodă.

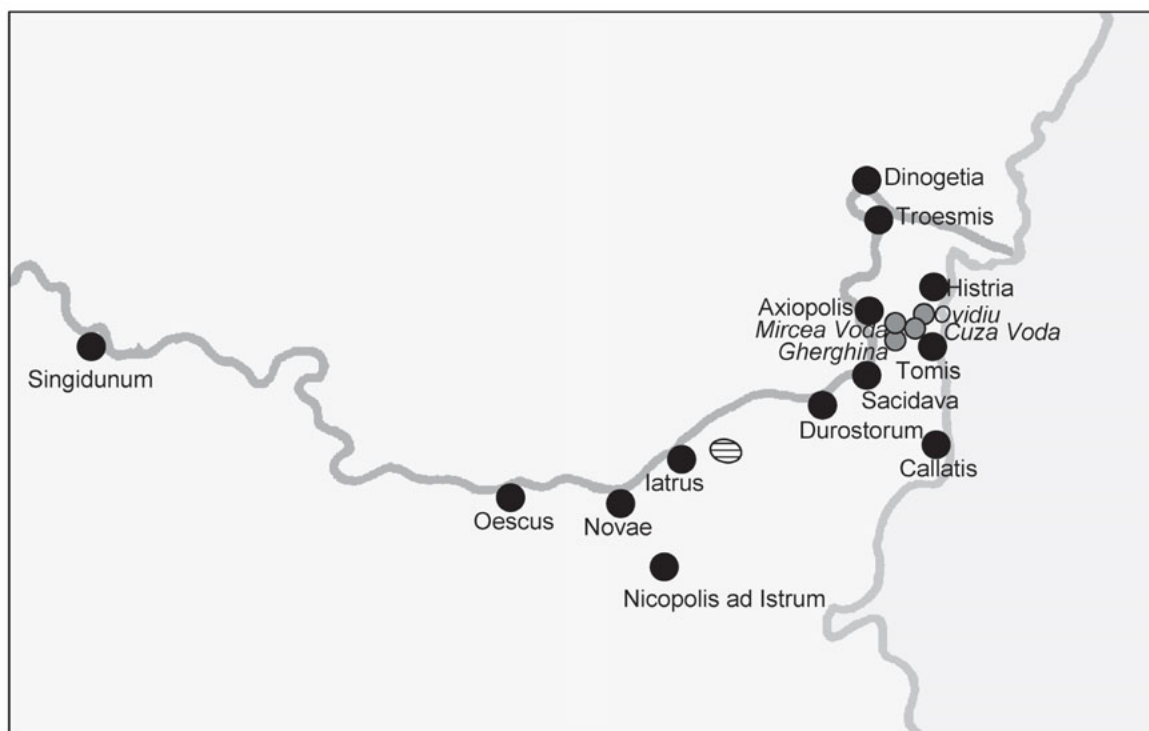
Multivariate cluster analysis (both for major elements and all samples of kaolinitic clays, and taking into account major and trace elements for only 11 samples of kaolinitic clays)<sup>16</sup> clearly demonstrates that LDKW 1–LDKW 6 pottery is totally distinct from the kaolinitic clays from Dobrudja. Only the sample representing group LDKW 7 is clustered with kaolinitic clays from Dobrudja (**fig. 7**). This is equally evident in the two-component diagram showing Na<sub>2</sub>O content versus CaO content. In this diagram the fragment of LDKW 7 pottery is separated from the remaining LDKW samples, which cluster within the LDKW 1–6 groups (**fig. 8**). The LDKW 7 ceramic sample distinctly differs in multivariate cluster analysis from the other twelve LDKW samples, in terms of temper composition (exclusively grains of quartz) and texture, as well as in the shape and pattern of its pores.

<sup>16</sup> Cluster analysis using Euclidean Distance and average link with the following elements: Si, Ti, Al, Fe, Mn, Mg, Ca, Na, K, V, Cr, Ni, Sr, Zr and Ba was carried out for all ceramic samples and 11 samples of kaolinitic clays for which trace elements were analysed; however for all ceramic samples and all 16 samples of kaolinitic clays cluster analysis was only carried out with the major elements Si, Ti, Al, Fe, Mn, Mg, Ca, Na and K (Brookhaven Data Handling Programs, kindly provided by E. Sayre). The results of both groupings show the same thing: that LDKW 1–LDKW 6 are completely different to kaolinitic clays from Dobrudja.



Sample number	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	V	Cr	Ni	(Cu)	Zn	Rb	Sr	Y	Zr	Ba	(Ce Pb)	LOI	TOTAL			
	% by weight										ppm											%	%			
a) LDKW found in <i>Novae</i>																										
LDKW 1																										
MD 3237	65,13	0,83	27,21	2,83	0,011	0,93	0,65	0,54	1,80	0,08	96	105	43	62	73	90	98	17	113	366	31	24	1,39	99,98		
MD 3238	65,11	0,83	27,22	2,83	0,010	0,93	0,64	0,55	1,78	0,09	100	116	42	62	72	89	98	17	114	374	39	32	1,34	99,71		
MD 3481	65,75	0,83	26,64	2,74	0,013	0,86	0,64	0,51	1,97	0,06	97	119	46	24	65	95	103	17	107	387	36	25	1,81	100,20		
LDKW 2																										
MD 2518	70,01	0,83	22,05	2,41	0,016	0,79	0,87	0,79	2,00	0,24	80	107	39	55	76	87	126	20	142	388	33	29	18,86	100,42		
MD 3235	68,58	0,75	23,57	2,50	0,018	0,82	0,89	0,80	1,96	0,12	95	94	42	78	69	82	109	17	118	437	39	21	1,48	99,56		
MD 3236	69,43	0,67	22,75	2,43	0,016	0,90	0,77	0,85	2,04	0,16	80	114	48	68	59	84	126	15	104	431	30	26	1,87	100,07		
LDKW 3																										
MD 2535	68,36	1,03	23,58	3,50	0,025	0,64	0,82	0,61	1,36	0,07	67	99	32	39	53	68	90	23	141	302	29	23	0,87	99,79		
LDKW 4																										
MD 3480	69,90	0,74	20,44	2,90	0,018	0,76	2,39	0,78	2,00	0,08	80	119	42	16	57	84	126	21	119	413	42	38	2,07	100,20		
MD 3482	70,03	0,75	20,61	2,93	0,018	0,73	2,06	0,81	1,99	0,08	90	107	44	16	55	86	124	19	129	436	42	28	1,74	99,80		
LDKW 5																										
MD 3479	71,38	0,95	19,81	2,21	0,017	0,72	2,25	0,71	1,85	0,11	74	98	45	27	51	78	117	22	147	416	40	24	1,98	97,35		
MD 3484	72,01	0,94	19,84	2,20	0,015	0,66	1,78	0,65	1,82	0,10	70	99	42	15	47	78	105	22	148	410	38	27	1,55	100,10		
LDKW 6																										
MD 2542	68,23	0,95	22,66	2,75	0,019	0,66	2,28	0,81	1,50	0,14	77	90	30	53	51	73	155	19	137	353	43	26	2,08	99,54		
LDKW 7																										
MD 3483	70,92	1,00	20,79	2,48	0,020	0,62	0,95	0,28	2,33	0,62	108	91	34	22	45	104	120	23	209	550	46	24	1,63	99,70		
b) kaolinitic clays from Dobrudja (Comşa 1985, 99)																										
	57-63	0,8-1,5	20-25	2-4,9		0,08-0,8			1-3,5 (Na <sub>2</sub> O + K <sub>2</sub> O)																	
c) kaolinitic clays from Dobrudja, S. Rădan																										
Cuza Vodă																										
SDR 33	62,94	1,30	28,60	2,71	0,011	0,85	1,11	0,29	2,17	0,01	133	79	23	<10			66		221	480		16	8,97	97,43		
SDR 36	65,85	1,10	24,84	2,52	0,011	1,37	1,54	0,23	2,54	0,01													6,07	97,11		
SDR 116	67,50	1,28	26,39	2,03	0,011	0,61	1,09	<0.1	1,07	0,01	131	72	33	44			61		233	380		9	9,29	99,35		
SDR 117	66,95	1,30	22,51	3,78	0,034	0,96	2,22	0,34	1,83	0,09	136	81	79	57			102		203	319		61	10,83	99,56		
SDR 118	61,96	1,49	24,67	3,61	0,011	4,14	1,85	0,23	1,95	0,09	152	113	37	76			150		196	403		67	12,63	99,68		
SDR 121	67,70	0,99	23,78	2,94	0,088	1,43	0,92	0,22	1,87	0,07	121	72	29	20			110		275	385		21	8,99	99,90		
SDR 122	60,21	1,02	29,35	3,12	0,102	1,59	0,95	0,33	3,27	0,06	158	136	25	40			122		199	478		20	11,44	99,69		
SDR 123	64,32	0,97	28,63	1,66	0,080	1,65	0,80	0,19	1,63	0,06	88	80	23	18			100		135	285		10	12,15	99,87		
SDR 127	59,40	0,96	28,08	4,91	0,000	1,71	1,55	0,21	3,12	0,07	166	101	29	55			161		157	445		30	14,20	99,97		
SDR 128	79,30	0,85	15,39	1,18	0,011	0,44	0,97	0,11	1,70	0,05	68	47	10	<10			76		360	297		7	5,05	99,56		
Gherghina																										
SDR 179	61,20	1,42	30,72	2,80	0,000	0,76	1,55	0,12	1,37	0,07	171	72	37	12			52		367	281		38	14,55	100,55		
SDR 182	76,17	0,95	17,22	2,22	0,032	0,78	0,74	0,10	1,71	0,08													5,13	99,78		
Mircea Vodă																										
SDR 73	74,76	0,75	17,09	3,15	0,000	0,64	1,35	0,26	1,94	0,06													5,54	99,16		
SDR 75	67,24	0,75	25,19	3,00	0,011	0,75	1,65	<0.1	1,39	0,01													6,10	99,75		
SDR 77	66,91	0,93	22,48	4,60	0,011	1,20	1,53	0,23	2,05	0,07													6,64	98,23		
Ovidiu																										
SDR 154	59,25	1,31	33,33	3,54	0,011	0,11	1,49	0,18	0,77	0,01	148	87	19	21			60		253	>1000		17	11,60	99,09		

**Table 3. a)** Results of chemical analysis by WD-XRF of LDKW. LOI = loss on ignition at 880°C; TOTAL = original sum before normalization to 100%. Values for S and Cl have not been included in these tables as, mostly they amounted to less than 0.01%. Trace elements determined with lower precision are given in brackets. **b)** Results of chemical analysis of kaolinitic clays given by M. Comşa, (COMŞA 1985, 99). **c)** Results of chemical analysis by S. Rădan, major elements normalized to 100%, original sum is given as TOTAL.



**Fig. 6.** Map of ancient sites and outcrops of kaolinitic clays. Grey point = analysed kaolinitic clays; striped area = outcrops of kaolinitic clay in vicinity of Ruse.

The comparison of chemical analysis results for LDKW pottery from *Novae* with those for kaolinitic clays from Dobrudja presented above reveals that only one ceramic fragment of the 13 analysed was made of the same raw material. As outcrops of kaolinitic clay in Dobrudja occur in the area between ancient *Axiopolis*, *Histria*, *Tomis* and *Sacidava*, it is within this region that the production centre in which the vessel representing group LDKW 7 would have been situated. Based on current research findings, the remaining 12 LDKW vessel sherds recovered from *Novae*, cannot be attributed to workshops located in Dobrudja. It is, however, possible that LDKW-type vessels were produced at local workshops in the *Novae* region using kaolinitic clays available in the vicinities of Ruse (unfortunately, the authors of this article have been unable to obtain any published results of analysis for these raw materials).

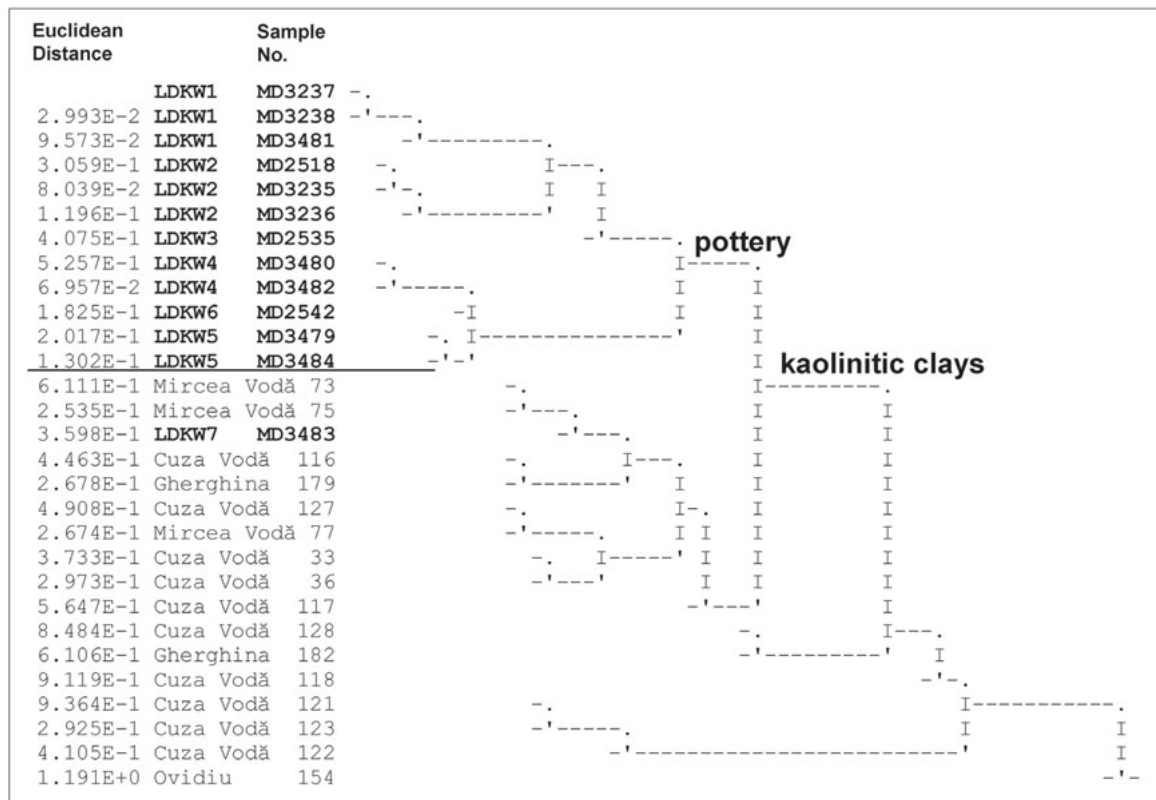
It cannot be said that the problem of where LDKW production centres were located has been resolved, particularly bearing in mind the results of chemical analysis of kaolinitic clays published by M. Comşa and those carried out by S. Rădan, as well as the analysis results obtained for 13 LDKW sherds found at *Novae*. There can be no doubt that workshops producing LDKW vessels were located within an area bounded by ancient *Axiopolis* – *Histria* – *Tomis* – *Sacidava*, though it must be taken into account that these may not have been the only centres making LDKW pottery. The optimism expressed in P. Dyczek's abstract and poster at the Cádiz conference was premature, as analysis results show that kaolinitic clays from the southern Dobrudja region were not the only raw materials used for LDKW ceramics. Further analysis is essential, both of raw material deposits from the

Ruse region and of LDKW ceramics, e.g. from *Singidunum* and sites in Dobrudja (ideally ceramic wasters from kiln sites)<sup>17</sup>.

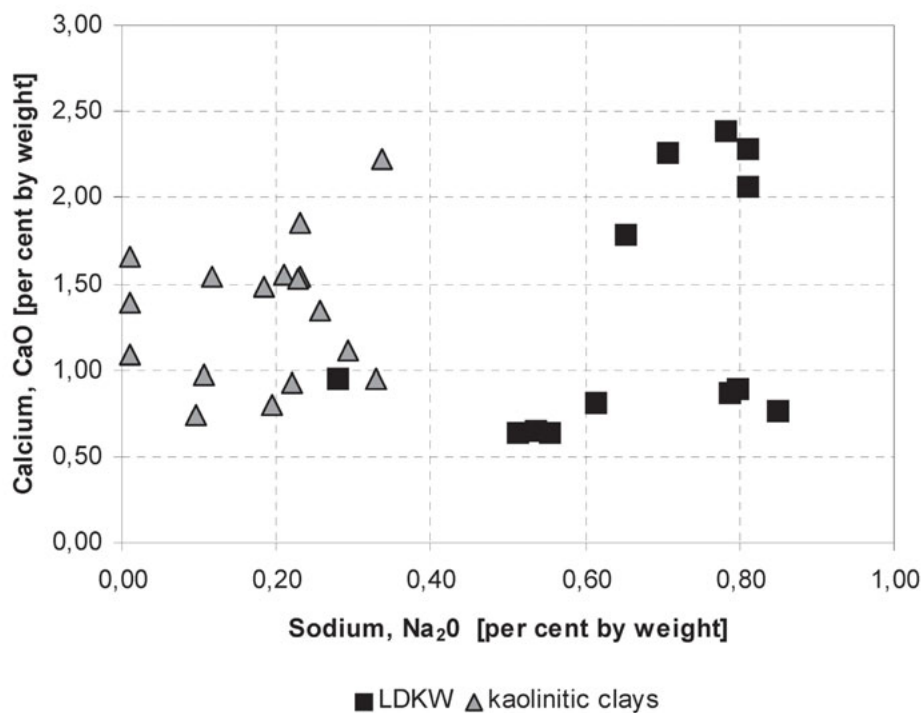
## Conclusions

1. All of the analysed LDKW fragments were made of clay raw materials containing kaolinite.
2. The colour of the pottery is linked to the content of iron compounds, which is high for kaolinitic clay deposits.
3. All of the analysed samples come from production areas having low Ti, Cr and Ni contents.
4. The analysed ceramic sherds were made at seven production centres (LDKW 1–LDKW 7), two of these production centres (LDKW 1 and LDKW 7) differing markedly from the others, both in terms of their chemical and mineralogical composition.
5. Based on the results of MGR-analysis nine groups of greatest similarity, pointing to a single workshop, can be identified.
6. The macroscopically visible differences in the texture and structure of the sherds are closely correlated with individual workshops.
7. Two vessels made at workshop 6 were produced from a single batch of clay, as was the case with two vessels made at workshop 7.

<sup>17</sup> To avoid possible errors due to different analytical techniques, the kaolinitic clays used for comparison should be analysed using the same technique.



**Fig. 7.** Dendrogram of cluster analysis using Euclidean Distance and average link with the following elements: Si, Ti, Al, Fe, Mn, Mg, Ca, Na and K (Brookhaven Data Handling Programs, kindly provided by E. Sayre). In bold are marked samples of LDKW.



**Fig. 8.** Diagram of sodium contents versus contents of calcium.



8. All of the analysed vessel's surfaces are unsmoothed and quite coarse due to the presence of non-plastic inclusions in medium sand fraction.
9. The vessels (or at least some of them) probably featured brown-red painted decoration.
10. The functional properties of LDKW pottery (based on analysis of four vessels) are appropriate for kitchenware vessels, i.e. for vessels intended both for storing liquids and for cooking (the analysed vessels are impermeable and resistant to thermal shock).
11. A comparison of analysis results for LDKW ceramic vessels with the results of analysis carried out on kaolinitic clays from Dobrudja reveals that only one fragment of pottery was made at a production centre in Dobrudja (LDKW 7).
12. Comparing analysis results for LDKW ceramic vessels with the results of analysis carried out on kaolinitic clays from Dobrudja demonstrates that twelve of the thirteen analysed LDKW vessels found at *Novae* were made from different kaolin raw materials than those known to occur in Dobrudja; these vessels were most probably made using raw materials that occur locally in the *Novae* region (kaolinitic clays from the Ruse area). Therefore, it cannot be said that the problem of provenance has been resolved; on the contrary, further analysis is essential, both of raw material deposits and of LDKW ceramics from other sites.

## Appendix

### Description of methods used

#### *MGR-analysis (Matrix Group by Refiring)*

Thin slices were cut from each sample in a plane at right angles to the vessel's main axis. One of these sections was left as an indicator of the sample's original appearance, whilst the remaining were fired in an electric laboratory chamber furnace, each one at a different temperature. Firing was carried out at the following temperatures: 1000, 1100 and 1200°C in air, static, with a heating rate of 200°C/h and a soaking time of 1h at the peak temperature. For selected samples refiring additionally was carried out at the temperatures 400°C, 600°C, 700°C, 800°C and 900°C.

#### *Chemical analysis*

Here chemical analysis by WD-XRF (Wavelength-dispersive X-ray fluorescence) was used to determine the content of major elements, including phosphorus and a rough estimation of sulphur and chlorine. It was also used to determine a series of fifteen trace elements, six of which, however, could only be ascertained with poor precision. Total iron was calculated as  $\text{Fe}_2\text{O}_3$ . Samples were prepared by pulverising fragments weighing *c.* 2g having first removed their surfaces and cleaned the remaining fragments with dis-

tilled water in an ultrasonic device. The resulting powders were ignited at 880°C (heating rate 200°C/h, soaking time 1h), melted with a lithium-borate mixture (Merck Spectromelt A12) and cast into small discs for measurement. This data is, therefore, valid for ignited samples but, with the ignition losses given, may be recalculated to a dry basis. For easier comparison the major elements are normalised to a constant sum of 100%.

The precision for major elements is below 1%, for trace elements this rises up to 20% depending on the concentrations. The accuracy is tested by analysing international reference samples and exchange of samples with other laboratories. For major elements and the most important trace elements it is between 5 and 10%.

Si = silicon, calculated as  $\text{SiO}_2$ ; Al = aluminium, calculated as  $\text{Al}_2\text{O}_3$ ; Ti = titanium, calculated as  $\text{TiO}_2$ ; Fe = iron, total iron calculated as  $\text{Fe}_2\text{O}_3$ ; Mn = manganese, calculated as  $\text{MnO}$ ; Mg = magnesium calculated as  $\text{MgO}$ ; Ca = calcium calculated as  $\text{CaO}$ ; Na = sodium calculated as  $\text{Na}_2\text{O}$ ; K = potassium calculated as  $\text{K}_2\text{O}$ ; P = phosphorus calculated as  $\text{P}_2\text{O}_5$ ; V = vanadium; Cr = chromium; Ni = nickel; Cu = copper; Zn = zinc; Rb = rubidium; Sr = strontium; Y = yttrium; Zr = zirconium; Nb = niobium; Ba = barium; La = lanthanum; Ce = cerium; Pb = lead; Th = thorium.

#### *Thin-sections*

Thin-sections were studied under a polarising microscope to provide some information on the matrix (the amount of information gleaned being dictated by the resolution of the microscope), primarily to estimate the composition and distribution of non-plastic inclusions. Quantitative analysis by point-counting was not carried out.

#### *Functional properties*

Functional properties analysis involved threefold measurement of water permeability rates. For analysis discs of 2 cm in diameter to be cut from sherds with both interior and exterior surfaces well-preserved. During each measurement the amount of water which penetrated the sample was noted after 1, 3, 6, 10, 20, 30, 50, 100, 200, 300, 400 and 1440 minutes. A record was also made of the times at which the following occurred: moistening of the exterior surface, the exterior surface becoming wet or water droplets seeping through the exterior surface. The samples were boiled in distilled water for two hours prior to analysis in order to remove secondary contamination from their open pores. Following this procedure water permeability analysis was carried out in controlled (hermetic condition): before and after heating at 400°C in air and after tenfold thermal shock at  $\Delta T=400^\circ\text{C}$  (using water as a cooling medium).

#### *Ceramic properties*

Open porosity, water absorption and apparent density were gauged using the hydrostatic weighing method.

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