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Summary

PHYSICAL-CHEMICAL ANALYSIS OF NEOLITHIC AND ENEOLITHIC CERAMIC MATERIALS IN BANAT AND TRANSYLVANIA

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Key words: pottery, Neolithic, Eneolithic, Archaeometry, FTIR, XRD, TG, SEM-EDX, XRF, LIBS

The general theme of the present endeavour is deepening the understanding of the prehistoric period, more precisely of the Neolithic and Eneolithic periods in Romania. The aim is applying physico-chemical analysis methods on material chronologically dated to these periods. In particular, the subject studied includes ceramic materials chronologically classified as Neolithic/Eneolithic from Banat and Transylvania, areas with numerous archaeological finds offering abundant material.

Specifically, the theme involves the study of materials from six archaeological sites from the mentioned periods, geographically located in the Mureș valley (middle and lower) and in the lowland Banat. Chronologically, the sites of Sânandrei-Oxenbrickel (com. Sânandrei, jud. Timiș), Șoimuș-Teleghi (com. Șoimuș, jud. Hunedoara), Iernut-Sit II (town Iernut, jud. Mureș), Foeni-Cimitirul Ortodox (com. Foeni, Timiș county), Ronaț-Triaj (mun. Timișoara, Timiș county) and Pecica-Est (Pecica, Arad county) cover the Late Neolithic and Middle/Late Eneolithic. The study of these periods in Romania is always a topical subject, with archaeological research being carried out continuously, leading to a continuous flow of new information, that often requires re-examination of previous results in the light of new discoveries.

The present topic is part of a (relatively) recent concern in recent decades to broaden the range of methods used in order to obtain the most conclusive and relevant results. We refer here specificcally to archaeometry, which includes methods of analysis and interpretation specific to other fields, especially those of the „hard” sciences. Unfortunately, the practice of using archaeometric methods is more widespread outside Romania, the reasons for this being multiple and not the subject of this paper. Following a preliminary analysis of the literature, it appears that our approach is in the direction of inter- and transdisciplinary research. It also follows the natural course of the author's previous work, concentrated on the analysis of archaeological materials using physico-chemical methods. Thus, the present work, once completed, complements the published works in the existing trends in the use of modern analytical techniques in general terms. In particular, the present approach follows other works which, cumulatively, provide an overview, albeit as yet an extremely limited one quantitatively but not qualitatively, of Neolithic and Eneolithic ceramic technology.

The aim of the present study is to obtain information as comprehensive as possible on the composition of the analysed ceramics, their provenance by correlating them with possible sources of raw material and estimating the firing temperature. Moreover, we aimed at outlining a working methodology in view of future approaches, both for us and for other researchers interested in its application. We therefore set out to identify the composition of the pottery at each of the investigated sites. Furthermore, we aimed to identify overall compositional characteristics, and according to ceramic vessel categories (fine, semi-fine, coarse). At the same time, we set out to identify differences, if identifiable, between categories, between ceramic species (where applicable), and fit these observations within the broader Neolithic/Eneolithic framework. The same approach has been taken with regard to firing temperature, by estimating it with as small a margin as possible, and then tracking differences between categories, sites and periods or cultural settings. The hypothesis from which we started was the possible existence of differences, observable in the composition and processing (again composition and firing temperature), both in space (geographical differences) and time (sites and materials occupy close but different chronological levels).

At the same time, we aimed to develop a comprehensive methodology for the objectives outlined above, based on the use of complementary, but also accessible, methods that allow us to obtain the maximum amount of information possible in relation to the objectives. The chosen methods are complementary, both grouped and sequentially. Furthermore, a methodology for recording the data after the analyses of the first sample batch (from Sânandrei-Oxenbrickel) has been defined, in order to fulfil a secondary objective, namely to outline a set of information that will serve as a starting point for outlining an incipient database on ceramic composition and firing temperature by region/period/cultural setting, to be used later by other researchers. The database remains a desideratum for the future, thus the option has been taken to publish the results through scientific articles, by the time the present work is completed, 50% of the results of the present endeavour have been published and the others are in the process of being published.

This work comprises three main segments. The first chapter forms the theoretical basis of the approach. We considered it appropriate to detail the physical-chemical analysis methods used. Each of these methods includes, according to the literature, details of the underlying principles and their use in the analysis of ceramic materials and a case study relevant to them. The order in which the methods are listed was intentional, as it is the order in which the analyses are carried out, firstly, and secondly, it is the natural order of their sequence within the approach. This is shaped by the characteristics of each analysis, starting with the simplest and then succeeding the analyses that directly complement the previous method. At the same time, the chapter includes a summary presentation of the main cultures present in Banat and Transylvania in the period in question (Starčevo-Criș, Pișcolt, Banat culture, CCTLNZIS/Zau, Turdaș, Herpaly-Salca, Foeni, Petrești, Ariușd-Cucuteni, Tiszapolgár and Bodrogkerestúr).

The second chapter covers the analyses carried out for each set of materials. Therefore, quantitatively speaking, this segment occupies a disproportionate position in the ergonomics of the work. The chapter is divided into two parts, one in which the sites of provenance of the material concerned are summarised, respectively their geographical and geological setting. The second part is in turn divided into 6 sub-chapters, each of which corresponds to one of the sets of materials analysed. Each of them include all the analyses carried out, the results in a preliminary state (primary processing on extraction from the apparatus with highlighting of relevant data), the description of the results for each sample, then a brief general conclusion for each set of analyses.

The third chapter contextualises the results and includes the interpretation of the results. The centralised results per location of origin were presented and then correlated with each other. In the last part, the contextualization itself took place, with the main historically relevant observations being traced, mainly concerning the degree of similarity of the pottery between the analyzed sites, then according to their chronological placement. The conclusions summarise the main results and their relevance both to the proposed objectives and to the wider framework of research in the field as a whole, and in particular to the method and period investigated. At the same time, the results have necessitated the outlining of some suggestions for future approaches, both by the author and by future researchers who may be interested in pursuing complementary approaches.

The analyzed sample batch from Sânandrei-Oxenbrickel consists of 12 ceramic fragments (fine, semi-fine, coarse) and 1 clay sample, that from Foeni-Cimitirul Ortodox consists of 19 ceramic fragments (fine, semi-fine coarse) and 4 clay samples, that from Ronaț-Triaj consists of 8 ceramic fragments (fine, coarse) and 2 clay samples, that from Iernut-Sit II consists of 7 ceramic sherds (fine, coarse semi-fine) and 1 clay sample, that from from Șoimuș-Teleghi consists of 12 ceramic sherds (fine, coarse semi-fine) and 4 clay samples, and lastly, that from Pecica-Est consists of 12 ceramic sherds (fine, coarse semi-fine) and 4 clay samples from Pecica-Est.

All sites are found to be located in areas of Quaternary deposits, formed over metamorphic, sedimentary and volcanic bedrock. Metamorphic rocks are formed as a result of modification of the existing rocks in a given geological deposit due to changing environmental conditions (temperature, pressure, mechanical forces, change in the chemical composition of the deposit), therefore they are predominantly found in active/inert volcanic areas, respectively in seismically active areas rich in eruptive and sedimentary rocks. Specific minerals include quartz, calcite, chlorite, kaolinite, muscovite, biotite, albite. At the same time, aluminium oxide-based compounds such as anorthite and spinel-like compounds, i.e. plagioclase feldspar and silica, are also found in such areas. Volcanic rocks are formed as a result of the cooling and solidification of magma at the surface, but magma also solidifies in the upper layers of the earth's crust. Volcanic rocks contain an abundance of minerals with a high content of silica (plagioclase feldspar, alkali feldspar), quartz, nepheline, muscovite or anorthite, and iron. Sedimentary rocks are formed at the earth's surface by the accumulation and lithification of sediments, formed by the physical disintegration and chemical decomposition of existing rocks, then the transport and deposition of these sediments to new locations. Sedimentary rocks include quartz-based minerals, calcareous minerals, feldspar, illite/montmorillonite and heterogeneous clay deposits, or loessososides.

For the Sanandrei-Oxenbrickel ceramics, the composition of the pottery has been established, with a high degree of uniformity observed, regardless of the category. FTIR analysis identified feldspar-like compounds (albite, orthoclase), together with silica, mica, montmorillonite, silico-aluminates, carbonates and quartz, respectively. The ceramics do not show clear distinctions between categories, but (minor but observable) differences were noted between the fragments in the 4 levels (in the case of coarse ceramics. Cer1 (5c), Cer10 and Cer11 (5d) show intense wavelengths for quartz, indicating higher content. Cer8(5b), Cer4 and Cer9 (5a) show more pronounced wavelengths for feldspar and montmorillonite respectively. This difference in fine ceramics indicates a different approach during the evolution of the settlement, indicating a higher quartz content, hence sand, in the earlier ceramic composites, and a lower one in the more recent sub-levels. Semi-fine and coarse ceramics invariably contain silica, mica and albite in composition, with wavelengths at similar intensities. At the same time, these two categories of ceramics show minor variations within categories, but also chronologically within each sublevel. The clay composition shows pronounced wavelength peaks for chlorite, analcime and attapulgite, which correlates, not very strongly, with the material analysed. At the same time, the presence of silica/quartz at low intensity wavelengths may suggest the use of high silica content admixture, together with the use of sand as a binder, in combination with the analysed clay.

XRD analysis confirms the composition, identifying within the ceramic samples pagioclase feldspars, albit, anorthite and muscovite (and/or illite), with a very good overlap in all samples. The identification of feldspars in different locations of the sherds (interior/exterior wall) indicates the use of a particular process of differentiation of categories during the manufacturing process. At the same time, the presence of spinel-like compounds in the fine ceramics indicates a thermal process applied at a higher temperature, indicating a qualitatively superior approach. The composition identified in the FTIR and XRD analysis fixes the maximum limit of the firing temperature, which did not exceed 800° C. Regarding the firing temperature, the thermal behaviour indicates a range of 550-650°C. Most of the samples show stabilization points for the processes occurring due to thermal degradation around the range 550-600°C. A slight increase in the estimated temperature is observed in more recent levels, with the maximum temperature for the first two sublevels being 550-600°C, whereas for the two more recent sublevels, the estimated burning temperature is in the range of 600-650°C. It is interesting to note that this variation is only noticeable within the sublevels, regardless of category, suggesting a qualitative increase as the settlement evolved.

For Foeni-Cimitirul Ortodox, FTIR and XRD analyses show the use of a ceramic paste with high plasticity caused by the presence of illithite and montmorillonite, which justifies the use of a quartz and mica-based binder.

Black ware contains albite and microcline, along with analcime, illite and montmorillonite. The binder consists of quartz and mica. The main crystalline phase identified for black ceramics is quartz, followed by phases for feldspar. It covers well the other phases present, namely albite, anorthite and illithite. The similar composition and crystalline phases are a strong argument for a similar clay source, most likely the same source. An albite and microcline based ceramic paste was also used for red pottery, together with analcime, illite and montmorillonite. The binder is also quartz, along with mica, less visible than in the case of black ware. At the same time, red ceramics contain more visible iron oxide-based compounds in all ceramic samples. The main crystalline phase is represented by quartz, together with albite, illite and anorthite. The similarities between the red ceramic samples indicate a common clay source for the raw material, but also common with that used in black ceramics. Similar to black and red ware, the composition of orange ceramics consists of albite and microcline, along with analcime, illcite and montmorillonite, with more visible iron oxides, similar to red ware. Quartz and mica constitute the binder. The main crystalline phase is quartz, feldspar (albite, anorthite) and illithite, together with mica. Crystal phases similar to those in black and red ceramics indicate the use of a common clay source as raw material in all three types. In regards to categories (fine, semi-fine and coarse), the differences in the crystalline phases indicate a different preparation method for the paste used to make fine and semi-fine ceramics, as opposed to coarse, with a possibly different thermal process for these categories.

Structurally, it is possible to differentiate between categories, but not by types, on the basis of homogeneity and porosity, with low homogeneity and high porosity for coarse ceramics, and high homogeneity and medium to low porosity for fine ceramics, and high homogeneity and low porosity (but not uniform) for fine ceramics. SEM-EDX identified 3 exceptions, F_CF 2 being of a noticeably better quality compared to the other pieces in terms of structure, and F_CF5 and F_CF7 (both fine, but from different types) being different compositionally, as they contain phosphorus. The composition of the clay shows a clear relationship between it and the ceramics, in particular the presence of plagioclase feldspar-type compounds (albite and microcline), together with analcime, chlorite and montmorillonite and magnesium sulphate heptahydrate, the main compounds in the samples analysed. At the same time, the presence of quartz explains the high proportion of silicon, beyond the use of quartz as a binder. The composition of the clay taken from the Foeni source, compared to that of the analysed pottery, indicates a high probability that the sampled clay was used as a base for the paste, and the particularities of each clay layer, taken together, fully explain the differences found in the samples. The only exception is the absence of illithe in the FTIR results, but crystalline phases for this mineral were identified by XRD analysis, which also identified in the clay all minerals observed in the pottery. In terms of SEM-EDX results, the clay samples are consistent in terms of the elements present in the ceramics, apart from titanium (not detected in this analysis but identified by XRF), and the proportions are consistent in 2 of the samples, although the use of a mixture of all 4 is a more plausible explanation. This argument regarding a local source of clay used in ceramics is confirmed by the analysis of the oxides present in the samples, including the clay, which in itself indicates the high probability of the use of clay from Foeni in the making of the analysed ceramics. On the other hand, differences between ceramic types are somewhat evident, with slightly different approaches for each species, but no significant differences can be seen according to category. The analysis of the elements present in the samples shows a difficulty in distinguishing on the basis of ceramic types, with the exception of the painted reddish-brown ceramics, with a similar but noticeably different composition. Slight differences are noticeable between fine and semi-fine ceramics, but more pronounced for coarse ceramics. At the same time, the presence of the same main and secondary elements in reduced quantities indicates a common source of clay for the paste of all the samples, the differences being explicable by different approaches specific to each category.

In terms of firing temperature, black ceramics show an estimated uniform temperature, with no differences between categories, in the range 550-600° C, with a maximum temperature of 600 °C, except for painted ware, with a slightly higher maximum temperature due to a secondary heat treatment for pigment stabilisation in the case of painted ceramics. The firing temperature is uniform for red ceramics and identical to that of black ceramics, estimated at 550-600° C, with the maximum temperature at 600° C. Exceptions are the painted ceramics, with 600-630° C in the pigment area, with a maximum temperature of 650° C, indicating a possible secondary heat treatment to stabilize the pigment. The clearest exception is the reddish-brown painted ceramic with a completely different thermal behaviour and an estimated firing temperature above 900° C. A uniform firing temperature at 550-600° C, with a maximum temperature at 600° C, is also observed for orange ware. The exception is the yellowish-orange pottery in the black-topped technique, fired in the black portion at a slightly higher temperature at 600-620° C, respectively one of the orange sherds with red painting with an estimated temperature for the pigment area of 550-630° C, with a maximum temperature of 650° C. At the same time, the thermal behaviour of the clay, specific for illite and montmorillonite clays, is similar to that of the ceramic samples indicates, an additional argument for the link between clay and the composition of the ceramic samples analysed.

The pottery from Ronaț was made from a ceramic paste based on plagioclase feldspar (minspar) compounds, together with analcime, illite and montmorillonite, as well as chlorite. This type of paste has high plasticity (caused by illithite and montmorillonite), which explains the presence of silica in the results, which has a very high content of quartz and mica, used as a binder. The similarities between the ceramic fragments suggest the use of the same type of paste for all analysed sherds. At the same time, the crystal phases, similar in all the analyzed samples, identified the presence of albite and anorthite. The similar composition of the sherds indicates the use of a similar, most likely the same, clay source. There are, however, differences in the crystalline phases, in relation to the categories (fine and coarse), the differences indicating a different mode of preparation for the paste used to make each category, and a different thermal process for them. The analysis of the clay indicates the possibility that it was used as a raw material, but the differences found, especially in the XRF analysis, do not make this possibility close to certainty, therefore, the use of this clay in the manufacture of ceramics can only be considered plausible.

Analysis of the chemical elements shows a difficulty in clearly differentiating between black, red and orange ceramic types, with a few exceptions that cannot be explained by belonging to different types. At the same time, the overlap in the electromagnetic spectrum can mask either the presence of silicon or aluminium, but they are necessarily present at the same time. Aluminium is also in visibly lower proportions in red ceramics. On the other hand, in terms of categories, there are slight differences between fine and coarse ceramics. But, with a few exceptions, the presence of the main and secondary elements respectively and in very small quantities is a significant indication of fingerprinting, indicating the use of a paste made from the same source with the same addition, the slightly different proportions either indicating specific approaches depending on the use of the ceramic or difficulties of detection in case of overlaps. Thus, the analysis of the elements indicates that the ceramic paste was made with at least a similar source of raw material.

Regarding firing temperature, black ceramics show an estimated uniform temperature, with no differences between categories, in the range 550-600° C, with a maximum temperature of 600° C, except for painted ceramics, with a slightly higher maximum temperature due to a secondary heat treatment for pigment stabilisation in the case of painted ceramics. The firing temperature is uniform for red ceramics and identical to that of black ceramics, estimated at 550-600° C, with a maximum temperature at 600° C. Exceptions are for painted ceramics at 600-630° C in the pigment area, with a maximum temperature of 650° C, indicating a possible secondary heat treatment for pigment stabilization. A uniform firing temperature at 550-600° C, with a maximum temperature at 600° C, is also observed for orange ceramics. The exception is the yellowish-orange pottery in the black-topped technique, fired in the black portion at a slightly higher temperature at 600-620° C, respectively one of the red-painted orange sherds with an estimated temperature for the pigment zone of 550-630° C, with a maximum temperature of 650° C.

Iernut ceramics are made from a tectosilicate (analcime), illithite and montmorillonite, as well as chlorite based ceramic paste. This type of paste has high plasticity (caused by illithite and montmorillonite), which explains the predominance of silica in the results, which has a very high quartz and/or mica content, used as a binder. The similarities between the ceramic fragments suggest the high probability of using the same type of paste for all analysed fragments. At the same time, the crystal phases, similar in the samples, show the presence of albite and plagioclase (anorthite) feldspar-type compounds. The similar composition indicates the use of a similar, most likely the same, clay source. There are, however, differences in the crystalline phases in relation to the categories (fine and coarse), the differences indicating a different mode of preparation for the paste for each category, and a slightly different thermal process for these categories. The analysis of the clay indicates the possibility that it was used as a raw material, but the differences found, especially in the XRF analysis, do not make this a definite possibility, therefore, the use of this clay in making the ceramics can only be considered plausible.

Analysis of the chemical elements shows a difficulty in clearly differentiating between black, red and orange wares, with a few exceptions. In terms of categories, slight differences are observed between fine and coarse ceramics. But, with a few exceptions, the presence of the main and secondary elements, respectively, and in very small quantities, indicates the use of a paste made from the same source, with the same addition, the slightly different proportions either indicating specific approaches depending on the use of the pottery, or difficulties of detection in case of overlaps. Thus, the analysis of the elements indicates that the ceramic paste was made with at least a similar source of raw material. Regarding firing temperature, black ceramics show a uniform estimated temperature, with no differences between categories, in the range 650-700° C, with a maximum temperature of 700° C, except for one piece (red ware, not painted), with a slightly higher maximum temperature. The temperature ranges of 650-700° C, with an estimated maximum of 700° C is applicable to all analysed types (with the exception mentioned above).

For the Șoimuș-Teleghi materials, FTIR and XRD analyses show the use of a ceramic paste with high plasticity caused by illithite and montmorillonite, a characteristic that requires the use of a quartz/mica-based binder. The fine ceramic contains analcime, illithite and montmorillonite, with a quartz/mica binder. The main crystalline phase identified for fine ceramics is quartz, followed by phases for feldspar, which covers the other phases such as chlorite, attapulgite and illite. The similar composition and crystalline phases are a strong argument for a similar clay source, most likely the same source. A ceramic paste based on analcime and chlorite was also used for semi-fine ceramics, together with illite and montmorillonite. The binder is also quartz, together with mica, as pronounced as in the previous category. The similarities between the fine and semi-fine ceramic samples make it difficult to differentiate between them, while indicating a common clay source for the raw material. The coarse ceramic is made from a similar paste based on feldspar-plagioclay compounds with illithite and analcime. Quartz and mica are again very pronounced in the composition. The two clay samples indicate a clear correlation with the pottery composition, with the same main minerals, analcime and attapulgite, but the use of a secondary source with illite/montmorillonite is inferred. The intense presence of silica and quartz partially corresponds to that in the ceramics, the partiality being explained by the later addition of the binder. We note the difficulty of distinguishing a differentiation by category.

In all samples, the main crystalline phase is represented by quartz, with a more or less pronounced coverage of the other phases. At the same time, the overlap is very good for pagioclase feldspars (albite, anorthite), and montmorillonite in most ceramics. Albite, with one exception, is present in all sherds, which makes an argument for the use of a common source, with a high proportion of quartz, beyond the binder used. Also, the lack of secondary phases is not conclusive, the low degree of crystallization as a result of a low-temperature firing making these phases difficult to detect, being necessary to correlate with FTIR analysis. The differences noticed between the categories are minor, due mainly to the variation in the amount of quartz used. The crystalline phases do not constitute an argument for the differentiation by categories regarding either the composition, or firing temperature. Correlation with FTIR results indicates a primary ceramic matrix composed of albite, analcime and quartz in all analyzed samples.

SEM-EDX results for both ceramics and clay confirm the composition based on ferric aluminium silicates, and the presence of calcium in undecomposed compounds confirms the low firing temperature, respectively an additional argument of correlations between ceramics and clay. At the same time, the only differentiation by category is based on the degree of homogeneity and porosity, with minor differences noticed from one category to another. Both oxide and element analysis results do not allow a major distinction between categories. At the same time, on the one hand, there is a slight distinction in composition, but also a phenomenon of masking aluminum oxides, respectively silicon, by category. This aspect indicates a different approach to processing, not composition.

The estimated firing temperature of the pottery is in the 550-600° C range, with a maximum temperature of 650° C. The firing temperature is not uniform for fine ceramics. Although the value of 650° C is the maximum, the fine black ceramic fragment shows slightly different thermal behavior, with a range placed at 600-650° C, and a temperature closer to 650° C is easier to argue for. The red sherd is estimated at a lower range, at 550-600° C, with the maximum temperature at 650° C being argued by much less obvious thermal processes. A similar situation is visible in the case of semi-fine and coarse ceramics, which show a uniform firing, at 550-600 °C, with the maximum temperature at 650 ° C being more plausible only for the red semi-fine ceramic fragment, and in the case of the coarse one, the estimated temperature does not exceed 600° C. The similar thermal behavior of clay is an argument for correlation with ceramics.

The Pecica-Est pottery shows a uniform composition, with a chlorite-based paste, with a very pronounced silicon-based component, both in the composition of the ceramic matrix and as a binder.

There are no noticeable differences between the categories, all being based on a paste very rich in silicon. The only difference is noticed in the composition of the coarse pottery, in which analcime and illite, as well as phosphates, were identified. The clay shows a similar composition with respect to the main minerals, but the limited degree of similarity indicates the use of a supplement in the composition of the pottery. Compositionally, quartz represents the main crystalline phase present in the samples, with good overlap for pagioclase feldspars (albite, anorthite), aith another crystalline phase present in all samples, being albite. Secondary phases are present in the form of anorthite in fine and semi-fine ceramics. The high degree of similarity regarding the crystalline phases indicates a common source for the ceramic matrix, as well as a crystallization resulting from a similar heat treatment. The observed differences are minor, caused by a variation in composition or firing, but insufficient to be categorized as intentional. At the same time, the presence of quartz and its degree of crystallization potentially mask other phases, insufficiently crystallized to be noticed. Structurally, all ceramic fragments have similar structures, with a low degree of homogeneity and relatively high porosity, with crystals of variable sizes in random groupings. The detected chemical elements are uniformly present, with a few exceptions, and the correlation between one of the clay samples and the pottery is strong. At the same time, the oxide analysis indicates a high probability regarding a common source of raw material for the basic ceramic matrix. At the same time, a different approach is visible depending on the category based on the traces/microtraces detected in a pattern that makes their unintentional presence unlikely. Furthermore, a differentiation can be observed regarding the elements, more precisely regarding the use of manganese, present in larger quantities in fine ceramics, then being present in smaller quantities in the following categories.

Viewed from an overall perspective, a uniform distribution is found in all the analyzed samples, regardless of category or origin, the presence of silicate type compounds, especially quartz, mica and silica, alongside a similarity regarding the clay from each site. This aspect must be seen from two perspectives. First, the clay source available at each site is sedimentary in nature, so it already contains high amounts of mica compounds (including illitic clays, from the hydromica group, and montmorillonitic clays, from the smectite group). Secondly, the composition of the highly plastic clay, typical of illitic/montmorillonite mixtures, which becomes brittle at high temperatures, must also be taken into account. Thus, pragmatically, it is necessary to introduce a binder with high hardness and low porosity, which binds the composition in order to undergo heat treatment, a fact that additionally explains the pronounced presence of quartz beyond the alluvial sedimentary character of the areas where the sites are located.

We find similarities between pottery and clay at each site in terms of the other minerals detected. However, while the specified minerals are generally found in the mentioned categories, it should be noted that variations within the category were identified in each site. Thus, we can state that there is a strong correlation, in this sense. At the same time, we note the uniform use of a ceramic paste based on ferric aluminium silicates, with a similar content, with a preponderance of chlorite and analcime, a phyllosilicate and tectosilicate respectively, from mineral groups found in similar geological layers. As we are discussing a different spatial and temporal range for the six sites, we cannot correlate a preference for an identical clay, the result not allowing this anyway, but we can correlate a preference for local clay sources in all six cases. The high degree of similarity between samples, regardless of category or site, is confirmed by the results of the XRD analysis. A uniformity is found regarding the basic ceramic matrix, composed of quartz, albite and anorthite. However, the same clarification above regarding variation within categories within the same sites should be noted here as well. The preferrence for using ferric aluminium silicates based clays, regardless of category of period, is confirmed in all samples by the XRF analysis.

No major differences can be distinguished between categories, with all samples containing considerable proportions of aluminium, silicon dioxide and ferric and ferrous oxides. The presence of calcium can be distinguished in all samples, except for those from Iernut. Thus, there is the possibility that the samples from this location were burned at a slightly higher temperature, associated with the decomposition of calcite, since calcium was identified in the clay sample. At the same time, the oxides present in small quantities, i.e. microtraces, coincide between clay and ceramics within each site, with small variations, which constitutes an additional argument for the local source of raw material. Noticeable differences are not identified even in the case of the elements, with aluminum and silicon being predominant, with the other elements being useful in tracing ceramics from a site. At the same time, an interesting phenomenon can be noted in the case of the fine pottery from Șoimuș (Vinča A3/B1), the coarse one from Ronat (Vinča C3/D1) and the semi-fine one from Pecica (late Eneolithic), in the sense that the aluminium masks the presence of silicon, clearly identified in the other analyses. We can issue the following statement, regarding the observation of ceramic composition in chronological evolution. Thus, in the middle Neolithic at chronological level Vinča A3/B1 (Șoimuș), in the Developed Neolithic, respectively Vinča B2 (Sânandrei), the Late Neolithic at level Vinča C/D

(Iernut, Foeni, Ronat), and the Eneolithic (Pecica) respectively, finds a relatively uniform use in the ceramic composition of a clay based on ferric aluminium silicates, the differences noticed being explained by those present in the local source of raw material. This relative uniformity must also be specified in relation to the location of the sites of provenance for the ceramics.

Regarding the firing temperature, the minerals present cannot provide satisfactory conclusions. Their presence, or diffraction intensity, is not conclusive, since the melting temperature of each one does not necessarily have a direct impact on the thermal behavior of the ceramic matrix. As a result of thermogravimetric analyses, corroborated with FTIR and XRD results, we estimate the firing temperatures to be in the range of 550-650° C. All samples, regardless of category or site, have a firing temperature that definitely exceeded 550° C.

The problem is about the maximum firing temperature. Thermal processes and mass losses were correlated with minerals identified in FTIR and XRD analysis, respectively. Thus, in the case of coarse ceramics, a high probability is found for a maximum firing temperature of 600° C. The appearance of the coarse ceramics analyzed (perhaps with the exception of Foeni), the minerals identified, as well as the thermal behavior of the samples indicate a slow, longer firing duration. At the same time, in coarse ceramics no transition phases are observed in the thermal behavior in specific intervals after 600° C. Semi-fine ceramics present a slightly different situation, being able to say in most cases that the minimum temperature exceeded 550-600° C, with a maximum temperature of 600-650° C, showing small variations within each sample. Fine ceramics, on the other hand, presents a more qualitative technological approach to firing, with a range of 550-650° C, and the maximum temperature is 650-700° C, as it presents slight thermal processes specific to the beginning of transition phases in minerals, exclusively in the stage preceding vitrification.

At the same time, in conjunction with the SEM analysis, we specify that the samples, with one exception, do not show the beginning of the vitrification stage, which excludes the 700-800° C interval. The correlation between the composition of the sampled clays, respectively the ceramic composition, indicates the presence of anorthite in the initial composition, which excludes its formation during the thermal process, formed after the decomposition of calcite in the range of 750-850° C. In the thermal analysis, the minerals present and their thermal behavior indicate the presence of incipient transition phases in the specific ranges (after 600 degrees), and the presence of quartz and the thermal behavior of the samples indicates a minimum temperature of 573° C when the first transition phase begins, but no subsequent phases are detected.

The identified composition is in accordance with other results from analysis carried out on Foeni ceramics. At the same time, the estimated firing temperature is within the range suggested in these results, although the methods used are only partially comparable. At the same time, the presence of ferrihydrite (iron phosphate) in the samples from Foeni indicates a maximum temperature of 650-700° C, the range in which its thermal decomposition begins, therefore this temperature was not exceeded. Local production is confirmed for Foeni, also established for Foeni type ware from Alba-Iulia Lumea Nouă. With regards to the Zau ceramics, we agree with the results obtained previously regarding the composition, respectively the firing temperature, although we would mention that the samples analyzed would rather fall within the lower limit of previously estimated ranges. Looking at the chronological evolution, we notice that in the middle Neolithic at chronological level Vinča A3/B1 (Șoimuș), in the developed Neolithic, respectively Vinča B2 (Sânandrei), in the Late Neolithic in level Vinča C/D (Iernut, Foeni, Ronat), the estimated temperatures are in the same general interval, which suggests a uniform approach both temporally and spatially. At the same time, a qualitative increase from one category to another is also observed. For the Eneolithic (Pecica), a lower qualitative placement is found, in relation to the previous sites, the firing temperatures, even in the case of fine ceramics, being placed towards the lower part of the range.

The preference for a relatively uniform ceramic composition and similar firing temperature, may be seen as a possible continuity with elements from the early/middle Neolithic, originating from the Starčevo-Criș culture, where similar characteristics are observed, both compositionally and for estimated temperature, as well as a relative uniformity in time and space and an affinity for local production.

CONCLUSIONS AND RECOMMENDATIONS

Following the completion of this work, we can draw some conclusions regarding its development. The use of the selected methods enabled the achievement of the objective of identifying the composition of Neolithic and Eneolithic ceramics.

We found that this was uniformly composed of ferric aluminium silicates, suggesting a common temporal and spatial approach to ceramic paste processing. Regarding the identified compounds, the ceramic paste consisted of a mixture based on albite/chlorite, analcime, mica, and illite/montmorillonite (or muscovite) respectively, which gives it high plasticity. This plasticity requires the addition of a binder with high hardness, a fact proven by the very pronounced presence of quartz in the composition. The composition was also determined by the geographical location, namely by the presence of sedimentary deposits, with very similar characteristics, in all the analyzed locations. We can see that the areas suitable for long term habitation, mainly river valleys, coincide with areas containing clay deposits suitable for the production of ceramics.

We can thus associate the composition for the pottery attributed to the Banat culture, the Foeni cultural group, the Zau culture (late phase), and the Tiszapolgár and Bodrogkerestúr cultures, respectively. Regarding the ceramics associated with the Middle Neolithic (Vinča A3/B), we maintain our reservations, the sample being small, and the cultural attribution for ceramics from the related site (Șoimuş) is preliminary. At the same time, considering the results obtained, we do not estimate a high probability for a future non-association, conditional on the utilization of a larger sample.

For the second objective, regarding the identification of the character of the ceramic production, whether local or not, we believe that we have established a strong correlation between the composition of the clay originating from the interior and the proximity of the targeted sites, and the composition of the ceramics. At the same time, we found that the differentiating elements between the samples from the sites coincide with the differences between the clays, relative to the location. Also, the differences within the sample batches, and within the categories, regarding the secondary elements, must be viewed with reservation, since it was found in the case of the Foeni ceramics that different elements were identified before and after the heat treatment. Thus, we consider that we have an additional argument for local production, as no case of possible import has been detected. At the same time, the fact that we have not identified any imports does not exclude their existence. Therefore, it should be noted that in relation to the batch size of the samples, any remark about imports should be correlated with future studies on more extensive samples from each site.

Compositionally, we have shown that there are no major differences between the sites regarding the categories of ceramics, a qualitatively superior approach being visible in the case of fine ceramics, which then decreases in the semi-fine and finally in the coarse. Thus, we are dealing with a uniform approach by category, observable from the middle to the late Neolithic. Regarding the Eneolithic (Tiszapolgár and Bodrogkerestúr), it must be stated that a qualitative approach with less pronounced differences between categories is noticeable. Since a clear differentiation by periods/cultures cannot be made with the results obtained, only those by locations being possible, we consider it appropriate to specify that classic methods, such as typological analysis, remain a necessity, the decorative elements and the more widespread forms being the only differentiating elements. Moreover, the high degree of specialization in the production of ceramics in the Foeni pottery is noticeable, where the red and orange ceramics show a higher iron content, which coincides, with a small margin of error, with the clay layers with a higher iron content, which indicates a deliberate use of a clay differentiated by the intended type.

Regarding the firing temperature, we believe that it can be placed, with a high degree of certainty, in the range of 550-650° C. This range is estimated for all samples, with one major exception, from all the sites concerned. There is a uniformity in time, from the middle to the late Neolithic, and space, both in Banat and in Transylvania, for this interval. The Eneolithic again presented a slightly special case, as it is qualitatively placed in the lower part of the range for all categories. Invariably, the maximum temperature for the coarse category is placed around 600°C, with few exceptions that warrant a higher maximum temperature estimate. Relative to semi-fine ceramics, the maximum temperature must be placed in the range of 600-650° C, with variations within samples without any particular pattern currently identifiable. Fine ceramics show, uniformly in time and space, a qualitative firing superior to the other categories, which led us to estimate a maximum temperature at 650° C, with possible exceptions reaching 700° C, with only one exception estimated as fired at a temperature above 900° C. The firing temperatures for the Eneolithic are qualitatively lower compared to the other samples, so the maximum temperature for this sample (Tiszapolgár and Bodrogkerestúr culture) does not exceed 650° C. A secondary observation should be offered regarding the painted, ware where the possibility of a secondary heat treatment is noted, probably carried out after the application of the painted decoration. Thus, we can associate the general range and the maximum firing temperatures for the ceramics attributed to the Banat culture, the Foeni cultural group and the Zau culture (late phase). We specify here that the Foeni cultural group contains the mentioned exception, originating from Foeni-Cimitirul Ortodox, with a firing at over 900° C. At the same time, we also mention here the aesthetically similar piece from Ronat-Triaj, which proved to be of inferior quality, which suggests the possibility of imitation, or the spread of the idea, but not the process (know-how) of implementation.

Considering the aforementioned, we consider that we have fulfilled the objectives regarding the composition, the source of raw material, the firing temperature, as well as the detection of any differences, in the sense that they are only recorded uniformly, spatially and temporally, between categories. Having said that, we indicate the need to expand the sample batch size, both quantitatively within each location, respectively, for a more conclusive picture, but also geographically, for a more comprehensive coverage. In this sense, this expansion of the sample size constitutes a promising direction of research in the study of prehistoric ceramics through physico-chemical investigation methods in general, and in particular regarding the period considered in the present endeavour. At the same time, we believe that a future contribution from other researchers is necessary to obtain relevant reference points regarding the results of the present work, as well as to obtain the most comprehensive data for Neolithic and Eneolithic ceramics.

Regarding the methodology, we consider that the objective has been partially achieved. First of all, the initial methodological approach, consisting of three methods, was confirmed as insufficient for obtaining the desired information, thus confirming some preliminary remarcs observed in the specialized literature regarding the physico-chemical analysis methods. The results for the first analysis were included in the present paper in the form originally obtained, precisely to show the limits found. The methodology required its gradual expansion by adding additional methods. Thus, the final methodology for our approach was outlined, which consists of a combination FTIR-XRD-TG-SEM/EDX-XRF-LIBS. We believe that this concatenation of methods, in the sequence mentioned, provides the most comprehensive and complete range of information regarding the composition of the ceramic, alongside the possible source of raw material and the correlation between them. At the same time, the specified methods show a high degree of complementarity regarding the estimation of the firing temperature, each of them providing information we consider partial, which cannot substantiate the firing temperature with a sufficient degree of certainty. Thus, their successive use and the correlation of the results provide an estimated interval as accurate as possible, limiting the margin of error specific to the singular use of a method, or a group of methods.

The use of the methods led to the following results:

- FTIR: allowed the identification of inorganic compounds, and mineral compounds (correlation with databases) respectively – the low homogeneity of the fragments partially distorts the results (undetectable compounds, lack of percentage)
- XRD: allowed the detection of crystalline phases of the main minerals – weak crystallization of some minerals/compounds partially distorts the results (undetectable phases, lack of percentage)
- TG/DTG: allowed the identification of the thermal behavior – the non-correlation with the FTIR and XRD analysis partially distorts the results (similar thermal behaviors between groups of unrelated chemical compounds)
- SEM/EDX: allowed the identification of sample structure, composition (elements) – non-correlation with FTIR, XRD, TG analysis partially distorts results/ wide error margin in results (erroneous estimates of firing temperature based on structure and composition)

- XRF: allowed the identification of oxides in the samples, detecting elements unidentifiable in the other results - non-correlation with previous analysis provides a low degree of precision (eg ferric aluminium silicates – wide range of possibilities)
- LIBS: allowed verification of other results by confirming elements/percentages – non-correlation with other results only provides preliminary/starting results.

Moreover, the redundancy of performing the analysis on the three areas (inner wall, outer wall and core/in section) was observed in the case of ceramics with the same color, without visible differences, as it does not change the results in any way.

Therefore, we consider that the methodological approach implemented by us within the present endeavour is viable for obtaining relevant results for the proposed objectives. But we specified that we do not consider that the objective regarding the methodology has been fully met. Although viable from a scientific point of view, the cost of the analysis carried out is prohibitive in the absence of collaborative networks between the institutions to which the archeology researchers belong and the institutions that have the necessary equipment and expertise to carry out these analyses. As such, it is a problem related to cost, collaboration and, at the same time, the availability of equipment, since the combination of equipment required for the methodology implemented by us is not available in a large number of research institutions in Romania, a fact that requires a collaboration between several institutions, which in turn implies a lower or higher degree of difficulty.

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