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PONDERING POTTERY – ELEMENTAL CHARACTERIZATION OF EARLY ENEOLITHIC POTTERY FROM ŠANAC–IZBA NEAR LIPOLIST

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XRF;
Early Eneolithic;
Western Serbia;
Pottery.

Abstract. Pottery from the site of Šanac–Izba near Lipolist was examined to reveal its chemical content. The site is located in western Serbia, Šanac–Izba near Lipolist. It was a small settlement only 40m in diameter and surrounded by a ditch. During the excavations in 2013 four pits and the remains of one burnt building were discovered. The site dates to the Early Eneolithic according to the regional chronology, late 5th millenium BC. Though three of the pits were of an unknown, yet later date. The pottery from the site is characterized by a diverse mix of cultural types that originate from the central Balkans and the southern Pannonian Basin. In particular, Bubanj and Tisapolgar culture influence is to be noted. Diagnostic fragments of pottery were sampled and analyzed for two key reasons. One, is there a significant difference in the chemical profile between the different pottery types? And two, can a chemical variability be observed between pottery that belong to different cultures? Forty one samples were selected based on their typology and in a way that represents all of the site features. Afterwards the samples were powdered and examined by pXRF and the results were statistically processed. According to the results, there is no significant difference in chemical profile between different pottery types nor is there a significant difference between different cultural traditions. Albeit, there was a single fragment of a storage vessel with calcium values that were several times higher than the average. Most likely this vessel (and its content) came to the site trough exchange, marital ties or as a gift.

Introduction

During the fifth millennium BC, great socio-economic and cultural changes took place in western Serbia and the central Balkans (modern-day Serbia) in general. These changes in demography and settlement size, population mobility and settlement pattern, economic strategies and cultural style also mark the end of Late Neolithic Vinča culture and the beginning of Early Eneolithic in the term of regional relative chronology (Tripković & Penezić, 2017, p. 1).

During the transition from the Late Neolithic to the Early Eneolithic, a new type of enclosed site emerged in western Serbia. This type of site was first described in the works of Trbuhović and Vasiljević (Трбуховић & Васиљевић, 1975) during the seventies of the twentieth century; the settlements are circular in base, surrounded by a ditch and are usually up to 50 meters in diameter (pp. 157–160). This type of settlement is primarily characterized by a ditch that serves as a defense against floods frequent in the wetland area of Mačva (Радовановић, 1994, pp. 8–10). Obrovac-type sites have been the subject of intensive study over the last decade.

In order to clarify the cultural practices of the inhabitants of the Obrovac type, it is important to perform archeometric analyses of samples from sites of this type. For this reason, this paper deals with pXRF analysis of ceramics from the Šanac–Izba site near Lipolist, which belongs to the Obrovac type. The aim of this paper is to enrich with another aspect not only interpretation of this site, but also to contribute to the understanding of the Early Eneolithic in general in the area of western Serbia.

Subject of Research

The site that this paper focuses on is Šanac–Izba near Lipolist in the Mačva plain (area of western Serbia), which is dated to the Early Eneolithic (Tripković, 2017, p. 53). The pottery of the Early Eneolithic period in northwestern Serbia is characterized by a mixed style of Early Eneolithic cultures, among which

the characteristics of the Bubanj culture (4500–3800 BC) are recognizable, as well as the cultures of Lenđel, Lasinja, Tisapolgar, and others (Tasić, 1979, pp. 97–98, 109).

Mačva was a very marshy area since the Pleistocene and presumably till the Middle Ages (Стојић & Церовић, 2011, p. 18) and is prone to flooding to this day (Stefanović et al., 2014, p. 86). According to the literature, the specific adaptation of the local population to such living conditions is reflected in settlements of the Obrovac type (Chapman, 1981, p. 117). Although most Obrovac-type sites are described as smaller hilly sites up to 50 m in diameter, there are also examples of extremely large settlements with ramparts. Most of the Obrovac-type sites were surrounded by a visible ditch, which was singled out as an important characteristic of the whole group (Трбуховић & Васиљевић, 1974, p. 160). Based on previous data, settlements of the Obrovac type can be dated to the fifth millennium BC (Трипковић et al., 2017, p. 75).

Site of Šanac–Izba near Lipolist

The Šanac–Izba site is located, about 1 km north of Lipolist (44°43'38.14"N, 19°30'4.81"E) in the Mačva plain. The village is located in the southern part of lowland Mačva, which is connected to the south by the hilly area of Pocerina and rises at the end to the mountain Cer. The pedological background of the site environment consists of pseudogley (Tripković, 2017, p. 51) characterized by stagnation of surface waters and moderate to high acidity (Aleksić, 2015, pp. 8–9, 32).

The site is in the shape of a smaller hill with a circular base, with a diameter of 35m to 40m, and at the highest point it is raised 1.5 m from the surroundings. A shallow depression resembling a ditch surrounds the site. On the south side there is a ramp about 2m wide that connects the hill with the area outside the village. This ramp was probably used to access the site (Трипковић et al., 2017, pp. 71–72).

Later magnetometric prospecting established that the site contained 1–2 buildings, which were significantly damaged by plowing and their remains were scattered throughout the site. Based on plant and animal remains, it can be concluded that a typical Neolithic-Eneolithic economy was practiced by the inhabitants. This is indicated by characteristic plant remains such as *Triticum monococcum*, *T. dicoccum*, *T. aestivum / durum* and in the case of animal remains *Sus scrofa domesticus*, *Bos taurus* and *Ovis / Capra*. Based on previous analyses of ceramic material and stone tools, as well as interpretations of stratigraphic records, it can be considered that this site was primarily occupied for a short period of time in the early Eneolithic (Tripković, 2017, pp. 53–68).

Two 2×2m trenches were investigated at the site, 4m apart, both in the central part of the site. Remains of at least one building and four pits buried in the pedological base were found in the trenches. The pits were measured

from a surface 2.0–2.5m deep. Yellow clayey soil is the pedological base in both trenches. This soil gradually acquired a darker shade in the higher levels, until it became distinctly dark gray, almost black. The average thickness of the dark layer was about 0.4m, but it was often much thicker. That dark layer was paleosoil on which cultural remains were deposited in the form of a collapsed building with other archeological material. Archaeological remains are covered with reddish-yellow sediment, which also contained small and medium-sized pieces of daub and ceramics. Above was a surface layer up to 20cm thick, devastated by plowing (Tripković, 2017, p. 72).

Excavation of two trenches revealed four pits and the remains of at least one dwelling. The floor of the dwelling was not found; however, fragments of oven flooring were sporadically found. Based on the vitrified pieces of daub, it is clear that the building burned at a very high temperature, and most of the ceramic fragments in the ruin layer also burned secondarily. Approximately simultaneously or somewhat later compared to the dwelling is pit 4. Pit 4 was noticed only after removing the destruction layer from trench 2, however, according to the reconstruction of stratigraphic relations, this pit was probably buried from the top of the destruction layer. Although further analyses are necessary to establish their relationship, pit 4 and dwelling 1 certainly date from the period of the original occupation of the site. Pit 4 differs significantly from other pits at the site in terms of filling, which in the lower half of the pit consists of thin alternating layers of ash-gray and red earth. Such a combination has not been found in the other pits (Трипковић et al., 2017, p. 66).

At least three pits (1–3) from the site are from a later period, however, the time of their burial cannot be determined with certainty. The origin of the sediment in the area of these pits can be traced all the way to the arable layer. The pits contained diagnostic pottery of Bubanj and Lasinja cultures but there is nothing in the site stratigraphy to indicate that the pits are the same age. Researchers rather tend to explain the situation as a secondary occupation of the site for an unknown reason. In their opinion, the pits were not backfilled after digging but were gradually filled through a process of natural sedimentation and erosion. Unfortunately, the details of the process of sedimentation cannot be easily reconstructed due to the fact that the upper levels of the cultural layer, approximately from the top of the destruction layer to the surface layer, may have been damaged in the past (Трипковић et al., 2017, pp. 66–68).

Pottery from the site is typical of Mačva and generally western Serbia during the early Eneolithic. Some of the most common types are beakers, conical bowls, bowls with a inverted rim, goblets with horn-like lugs and bowls with horn-like lugs, larger forms such as amphorae, etc. Various parallels can be found with these forms at sites that are spatially and temporally close, such as Livade–Kalenić and Bodnjik (Благојевић, 2005, pp. 50–52; Живановић, 2013, p. 17).

Research Goals and Hypotheses

In this paper, the current knowledge about the Šanac–Izba site is enriched by applying XRF analysis to selected ceramic samples using a portable device. This analysis proved to be suitable because the method itself is characterized by high speed of data acquisition, low destructiveness and good quality of analysis (Papachristodoulou et al., 2006, p. 347).

This paper has two main analytical steps. The first step was to examine the similarities and differences in the chemical compositions of different types of vessels, as well as the correlation between the type of vessel and their inclusions and fabric. There are studies that have addressed the issues of the operational chain and paste recipes in the Vinča culture in Serbia (Amicone et al., 2020; Perišić et al., 2016; Spataro, 2018), but such analyses have not been performed on Eneolithic material from western Serbia.

In this paper it was assumed that there is a difference in composition between different types of vessels, i.e., that they used different paste recipes for different types of pottery at Šanac–Izba. The hypothesis of a difference in composition between different types of vessels was based on the assumption that vessels with a rougher fabric contain a larger amount of non-clay elements (Rice, 2005, p. 324).

Of course, we should not forget how much the composition of the ceramic paste can vary (even within the same vessel) according to its chemical content, for which there are several reasons. It can be assumed that all ceramics from one Neolithic site will be similar, because in the Neolithic, local clay and inclusions were primarily used to make pottery. However, this may not be the case at all. Ethnoarchaeological research of Pereulele pottery in Spain is well known, which showed that pottery, although produced from local clay (even by the same potters), had a statistically significant difference in composition between vessels (Buxeda I Garrigos et al., 2001, pp. 2–11).

The variability of clay in the paste provides data on the preparation of raw materials and their origin. In ethnographic studies dealing with the production of ceramics, potters choose raw materials based on several factors, among which the properties of the material and ease of acquisition stand out. However, the choice of clay for making ceramics can also be influenced by taboos and rituals. Added inclusions are easily recognizable if they are not naturally present in the clay (e.g., bone, slag, large pieces of grog), while for those that may be naturally present, such as sand, it may be difficult to say whether they were added by a potter (Amicone et al., 2020, p. 89).

The second step of the research was to test the origin of vessels with horn-like lugs, which are an important characteristic of the Eneolithic in western Serbia and are very common in the area north of the Sava and Danube. A certain part of the ceramic assemblage of the Šanac–Izba site consists of vessels with horn-like lugs. Previously, this type of handle was considered to be an

influence from Pannonian Basin, and the presence of such vessels indicates a certain degree of influence of this area on Mačva (Живановић, 2013, p. 27). Examining them will reveal which paste this type of vessel is made of, and thus whether they were made in another way compared to other types or not. It was expected in this research that ceramics with horn-like lugs would have similar if not the same chemical content as the other types.

Materials and Methods

The pXRF device used is a Niton XL3t GOLDD + manufactured by Thermo-Fischer Scientific (2010). The detector type is a geometrically optimized large area drift detector. The instrument has a silver anode with a voltage of 50kV and 200μA. The pXRF instrument was calibrated to obtain usable data. There are many ways in which a pXRF device can be calibrated and some methods are more and some less accurate. However, even with the best calibrations, there are differences in the concentrations of many elements, depending on the reported study, from 2% to as much as 36% (Conrey et al., 2014, p. 298). It should be noted that in this paper, the goal is not to achieve the exact composition of the sample, i.e., real quantitative analysis (nor is this possible with the pXRF device), but to find a general elemental trend. For calibration purposes, a reference sample (XL3t catalog number 195–019) was used, as well as soil and ore testing standards from the ThermoFischer manufacturer (XL3t Soil / Mining QC 500–705; Reference Sample Set).

The spectra obtained by pXRF analysis were then statistically processed in the SPSS and Orange programs. Primarily cluster analysis, correlations, and principal component analysis (PCA) were used. Differences in composition were considered statistically significant for r values less than 0.05, ie., less than 5% probability that the difference is the result of a chance. This value is standard for most statistical analyses.

A sample of 50 sherds of pottery was used to analyze the pottery from the Šanac–Izba site. Samples were taken in such a way that most units and all features were represented. Diagnostic fragments (rims, handles, bottoms, etc.) were used for the samples for comparative stylistic-typological analysis.

The problem with pXRF analysis is that the data on the composition of the sample, especially in samples such as ceramics, are subject to the influence of difficult to control experimental conditions such as porosity and geometry of the analyzed ceramics. Particularly lighter elements are at risk of analytical errors in samples that have an abraded, irregular surface (Gauthier & Burke, 2011, p. 284). Thus, samples were prepared by powdering the pieces of pottery. A sample weighing about five grams was removed from the sherds. This powder was then transferred to an XRF powdered sample box and covered with

plastic wrap. Powder samples were prepared in a fume hood at the Faculty of Pharmacy in Belgrade to eliminate excess dust that could contaminate other samples. Between the preparation of each sample, the mortar and the pestle were wiped with a disposable paper towel and distilled water (Fig. 1).

Figure 1. Diagram of sample preparation

Although certain authors perform two or three measurements per sample in order to then use the mean but, this usually does not lead to significantly different and/or better data. However, this is not always the case (Liritzis et al., 2020, p. 8). For this reason, two measurements of each sample were used in this study. In the statistical analyses themselves, only the means of these measurements was used.

All samples were analyzed in soil mode for at least 105 seconds. Of these, 60 seconds was used in the filter for basic elements that make up the majority of the sample composition, 30 in the filter for trace elements with a low atomic number, and 15 seconds in the filter for trace elements with a high atomic number. These measurement lengths are considered sufficient for quality analyses on the pXRF device used, although longer measurements such as 60 seconds in each filter can be used, but no significant accuracy improvement is obtained (Thermo Fisher Scientific Inc., 2010, p. 151, 163).

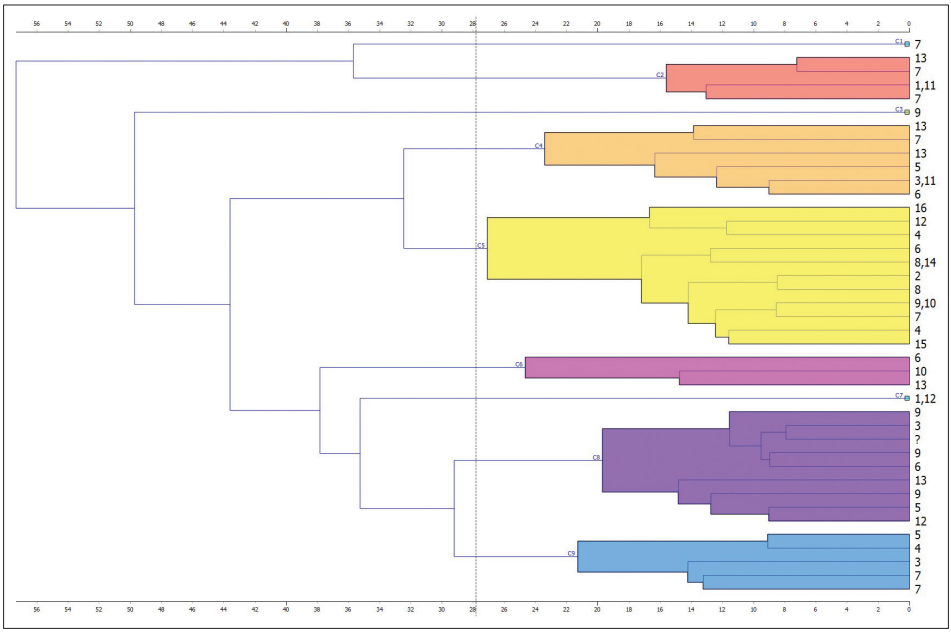
Before the statistical analyses, the data was, of course, standardized. This is a necessary step in statistical data processing where the data points do not have the same lower and upper limit. After that, descriptive statistics were made for all samples. All statistical analyses were performed in the SPSS (version 22) and Orange (version 3.28.0) program for statistical data processing. Most of the data visualizations were made in the Orange program.

Results and Discussion

In total, 33 elements were analyzed: Fe, Ca, K, Mn, Ti, Mo, Zr, Sr, U, Rb, Th, Pb, Au, Se, As, Hg, Zn, W, Cu, Ni, Co, Cr, V, Sc, S, Ba, Cs, Te, Sb, Sn, Cd, Ag, Pd. According to the results, Fe, Ca and K make up the largest share in the composition (above 10,000ppm), the amounts of Mn and Ti are also significant (below 1,000ppm), and all other elements can be found in traces (below 500ppm). It should be noted that all these elements are impurities of the clay itself, which primarily consists of aluminosilicates.

Samples were analyzed first to find out if there was a correlation between chemical composition and ceramic types (Fig. 2). Based on hierarchical clustering of all analyzed elements with Manhattan distance and Ward linkage, there are nine clusters in the Orange program, two of which are individual samples. No clear groupings around different ceramic types can be observed here, but all types can be found mixed in clusters. The annotations in the Figures 2 and 3 represent pottery types: 1 – conical bowl, 2 – biconical bowl, 3 – bowl with inverted rim, 4 – bowl with lug like handle, 5 – beaker, 6 – foot of a beaker/conical bowl, 7 – beaker with two handles from the rim, 8 – amphora, 9 – rough vessel presumably used for storage or cooking, 10 – strip like handle, 11 – lug like handle, 12 – tunnel like handle, 13 – pot base, 14 – ornament.

Figure 2. Hierarchical clustering with all elements



One of the performed statistical analyses dealt with the correlation of exclusively calcium (Fig. 3).

Figure 3. Hierarchical clustering with only calcium

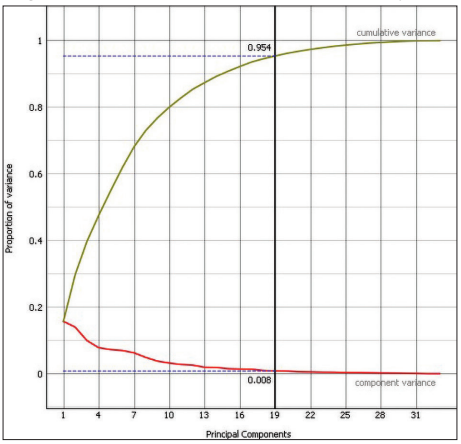
Two clusters were formed, the second of which contains all samples except one, a sample named 20-38-2. It is even more unusual that 20-38-2 is a fragment of a large vessel (9) that has very few visible inclusions and with a fine fabric. This means that the high calcium content is not the result of inclusions but the use of clay that is naturally rich in calcium carbonate (Vuković, 2017, p. 27). Thus, the sample 20-38-2 was either an experiment using another source of clay, a fluke, or perhaps was even brought in from somewhere. Why a different source of clay would be used for only these vessels alone cannot be known with certainty. It is unlikely that the clay deposit used to make all the other vessels became inaccessible, because the site was not inhabited for a long time. There is a possibility that the vessel reached the site by exchange from a nearby site from the Early Eneolithic, such as Šanac in Obrovčine, Majur–Veliki Obrovac, and others. Why a different source of clay would be used for this one vessel alone cannot be known with certainty. It is unlikely that the clay deposit used to make all the other vessels became inaccessible, because the site was not inhabited for a long time. It could possibly be a consequence of the blurring of differences between populations and cultures that is characteristic of the region during Early Eneolithic. There is also the possibility that the vessel reached the site by exchange from a nearby site from the Early Eneolithic, such as Šanac in Obrovčine, Majur–Veliki Obrovac, and others.

The second analysis deals with fabric, inclusion size, and their amount. Of the four clusters discovered, the second of which contains most of the samples, while the first cluster consists of a few diverse (inclusion and fabric wise) members. Along with those two there are two single member clusters and one

of them has a fine fabric (sample 30). It is clear that all samples are quite mixed in the clusters when it comes to inclusions and fabric which confirms that there is no correlation between inclusions, fabric and chemical content.

PCA was also performed, i.e., analysis of principal components (Fig. 4).

Figure 4. Principal component analysis



In the analysis of the samples, 19 elements explain 95% of the variance. In any case, a large number of elements contribute to the variance, and the way clusters are formed cannot be based on only a few characteristic elements. There is no noticeable grouping by ceramic categories by principal components.

Louvain clustering was performed together with the analysis of principal components and two relatively close clusters were formed. These clusters do not appear to be formed on the basis of differences in ceramic type or the type and number of inclusions. Within 2 standard deviations from zero, 66% of the samples are present, which means that there is a significant number of samples with unusual values. What should be noted are the two samples that represent the outliers of the first and second clusters sample 21 (cluster 1) and sample 30 (cluster 2). Sample 30 is a fragment of a large vessel. Sample 21 is not yet reliably typologically determined. Both samples have very high values of principle component 2 but differ completely in the amount of principle component 1 where Sample 30 is six positive standard deviations away from zero and Sample 21 is four negative standard deviations away from zero.

According to previous analyses, there is no statistically significant difference in the chemical composition of vessels between types. This means that the locals did not use different paste recipes for different types. Different recipes of the paste would not have been used if there was no real need for them (Vuković, 2017, p. 53). Samples of vessels with horn-like lugs can be found scattered in clusters, which means that there is no statistically significant difference in composition between this type and others.

Conclusion

Ceramics from the Šanac–Izba site were examined with the primary goal of determining variations in the composition of ceramics. The first step was to determine whether there was a difference in the compositions of the vessels between the different types of pottery at the site. The second step dealt with the vessels with horn-like lugs because this form was previously considered an influence from the Pannonian Basin, and the question was whether this type was made from the same recipe as other forms or different. This information is important because it sheds light on the technology of making ceramics at the Šanac–Izba site.

Ceramic samples have been analyzed with the pXRF instrument, a method that has been widely and successfully used in archeology for decades. This instrumental method was chosen because of its satisfactory accuracy, speed of data acquisition and affordability.

XRF analysis found that there is no significant difference in composition between different types of vessels when all the analyzed elements were taken into account. This indicates that the inhabitants of Šanac–Izba used a 'universal' paste recipe for different functional types. The only confusion is the sample 30, which contained a much larger amount of calcium. The reason for this cannot be reliably determined without additional analyses. Possible reasons are, first of all, exchange and use of another source of clay for an unknown reason.

No statistically significant difference in vessel composition was found between different fabrics and inclusions. The division into ceramic types of rough and fine fabrics was observed in other localities that are temporally, spatially and culturally close to the Šanac–Izba site, such as Livade–Kalenić, Bodnjik, and others. It is not yet known whether there is a difference in composition between the different fabrics and inclusions at the above-mentioned sites. Vessels with horn-like lugs were shown not to have a statistically significantly different composition compared to other vessels. This means that this category of pottery was not imported but was produced from the same raw materials as other types of ceramics. This result was expected, considering that local clay is most often used in the Neolithic, and the import of ceramic vessels with their content was at a low level during the early Eneolithic.

Despite the fact that this study did not lead to too many unexpected conclusions, it confirmed certain hypotheses. The only surprise is the result that there is no difference in composition between different types of pottery.

It should be noted that these samples were tested with a pXRF device that has difficulty to detect nuances in the composition in contrast to techniques such as FTIR, INAA, ICP-MS and others (Tykot, 2016, p. 46). Therefore, these preliminary results should be taken as incomplete. It would be useful in the future to improve the research by testing these samples with another useful

and complementary method, such as FTIR, NAA, ICP-MS, and others. In the absence of the above-mentioned methods, it would be necessary for further research in this direction that the samples be subjected to microscopic analysis in order to identify minerals in the samples. This analysis is a good starting point for pXRF analysis of pottery from several Obrovac sites. Sampling of not only pottery but local clays as well, should be considered in order to identify potential clusters in terms of recipe and sources of raw materials used. Gajić-Kvaščev & al. (2012) for example, managed to examine Neolithic pottery from several sites by pEDXRF analysis and then used this data to classify unknown pottery samples (p. 2).

Obrovci are an important phenomenon characteristic of Mačva and the period of transition from the late Neolithic to the early Eneolithic. However, there are not many papers dealing with the problem of Obrovci, because for a long time this type of site saw little attention due to the assumption that they are related to trenches from Serbo-Turkish wars. Therefore, knowledge about Obrovac-type settlements is limited by a small number of excavations and research. However, the situation is starting to change for the better lately and there is a noticeable increase in the interest of the archaeological public for localities of the Obrovac-type. Unfortunately, the transition period between the Neolithic and the Eneolithic in the central Balkans is little researched and equally unknown. We hope that future research of Obrovac-type sites will lead to new knowledge about the time when the Balkans, as always, was experiencing a great series of socio-economic changes.[2]

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Резиме

Током петог миленијума п. н. е. дошло је до великих друштвеноекономских и културних промена у западној Србији, као и на централном Балкану уопште. Ове промене најавиле су крај позног неолита и винчанске културе, али и почетак раног енеолита у релативној хронологији. Током транзиције из позног неолита у рани енеолит јавља се нови тип насеља зван Обровац у западној Србији. Ове насеобине кружног облика оивичене су јарком и пречник им је најчешће до 50 м. Јарак је овим насељима био кључна карактеристика и служио је као одбрана од поплава које су биле врло честе у западној Србији, а нарочито у Мачви.

Ради разјашњења културних пракси житеља Обровца, било је неопходно извршити археометријске анализе узорака керамике са једног таквог локалитета. Керамика, као најчешћи налаз на археолошким локалитетима помоћу метода природних наука, може пружити обиље података о животу у прошлости. Из тог разлога, овај рад бави се анализом керамике са локалитета Шанац–Изба код Липолиста, који припада локалитету Обровац. Циљ овог истраживања није само употпунити интерпретацију овог локалитета већ и допринети разумевању раног енеолита уопште у западној Србији.

На локалитету Шанац–Изба откривени су остаци једног објекта и четири јаме. Објекат и једна јама датују се у рани енеолит, док су остале јаме накнадно ископане. Керамика са локалитета представља мешавину културних утицаја са централног Балкана и јужног дела Панонске низије. Узет је 41 узорак од

дијагностичких фрагмената керамике који представљају све целине са локалитета да би се одговорило на два главна истраживачка питања, и то: Да ли постоји значајна разлика у хемијском саставу између различитих типова керамике? Да ли постоји значајна разлика у хемијском саставу између типова који припадају различитим културним традицијама? Узорци су након одабира били спрашени те анализирани. Анализа је извршена XRF аналитичком методом, а на крају су добијени спектри статистички обрађени.

На основу резултата не постоји статистички значајна разлика у елементарним профилима између различитих типова, односно, функционалних група, као ни између различитих културних група. Керамика на локалитету, технолошки гледано, прављена је на различите начине, али од истих сировина. Међутим, треба напоменути један необичан узорак који потиче од посуде највероватније коришћене за дуготрајно складиштење намирница. Овај узорак садржи многоструко веће вредности калцијума у односу на остале узорке и то не може бити случајно. Фрагмент ове посуде највероватније је завршио на локалитету због размене или као поклон из другог насеља. Локалитети попут Обровца нису довољно истражени, као ни период раног енеолита у Србији, али ситуација се мења набоље у последње време и надамо се да ће се у будућности овај позитиван тренд и наставити.

Кључне речи: XRF; рани енеолит; западна Србија; керамика.

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