The reconstruction of functional zones at Neolithic to Early Iron Age sites in the Neva river basin (Russia) by means of geochemical markers

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Abstract

Anthropogenic activity by ancient people influences the chemical composition of soils, enriching or exhausting certain chemical elements. The application of geochemical methods allows us to obtain important information for interpreting functional zones at settlements (for example, places of habitation, cooking zones, fireplaces, and so on). Such research was conducted at two sites dating from the Neolithic to the Early Iron Age in the Prinevsky region – Okhta 1 and Podolije 1. Complexes of geochemical indicators connected with certain functional zones of anthropogenic activity were identified. The burial place at the Okhta 1 site is marked by abnormal values of the bioindicators P_2O_{5antr} and CaO_{antr} . The geochemical complex at the Podolije 1 site, including indicator ratios of K_2O_{antr} , Rb_{antr} , CaO_{antr} , and Sr_{antr} , can characterize fireplaces (K_2O_{antr} and Rb_{antr} make up a part of ashes) and zones of fish and animal preparation (CaO_{antr} and Sr_{antr} are elements of bone tissues). Such investigations have been conducted at only a few archaeological sites in the territory of Russia, and this research is unique.

Keywords: geochemical anthropogenic indicators, environmental reconstruction, archaeology, Okhta 1 site, Podolije 1 site, Lake Ladoga, Stone Age

Introduction

At present, different scientific methods are used at archaeological excavations to obtain more information about the palaeoenvironment, the period of habitation, and other features of ancient human life. One important task is the reconstruction of functional zones at ancient settlements. Sediments and soils affected by anthropogenic activity reflect this information in their physical and chemical characteristics. Valuable information about anthropogenic activity at settlements can be obtained from the geochemistry of soils and cultural deposits. The activities of prehistoric people influenced the variations in the chemical compositions of the soil by enriching it with or depleting it of certain chemical elements, ultimately creating that which constitutes archaeological soils and cultural layers (Oonk et al. 2009). High concentrations of heavy metals in the soils of archaeological sites correlate closely with the anthropogenic activity of ancient people (Aston et al. 1998; Entwistle et al. 1998; Wilson et al. 2008). This can be explained by the fact that the geochemical markers of anthropogenic activity are conserved in the deposits for many years.

The most frequently used chemical elements for the determination of anthropogenic activity at ancient sites are P, Ca, K, Na, and Mg, and the microelements used include Cd, Cr, Cu, Pb, and Zn (Lutz 1951; Aston et al. 1998; Schlezinger & Howes 2000; Terry et al. 2004; Cabala et al. 2012). If the archaeological sites are located in areas with complex relief, the primary distribution of chemical elements may have changed as a result of erosion after the elements were originally deposited in the soil. Soil erosion processes connected with the effects of water, wind action, and ploughing redeposit the geochemical components downhill. The destroyed material accumulates in depressions and at the feet of slopes as colluvium. Research at this kind of archaeological sites with complex relief was described by Oonk et al. (2009). These cases require the development of a uniform methodological approach, the determination of a precise geochemical background, and the understanding of geochemical processes at the site. Nevertheless, even in complex geomorphological contexts, anthropogenic geochemical indicators can be used to reconstruct functional zones at ancient settlements.

Aston et al. (1998) considered the connection between high concentrations of heavy metals in soils and ancient anthropogenic activity. High concentrations of heavy metals can be connected with different types of ancient anthropogenic activity as a result of which chemical elements of anthropogenic origin have accumulated in soils. For example, these elements may indicate the development of ancient settlements, animal breeding in close quarters, the use of fire (fireplaces, slash-and-burn cultivation), ancient metallurgy, or subsistence activities (production of leather, processing of agricultural crops). As Schlezinger & Howes (2000) suggest, increased phosphorus concentration in soils can be connected with the physiological activity of humans and animals in their habitation areas, the decomposition of animal and plant organisms in settlement areas, and the use of animal dung as fertilizer. Several researchers (Terry et al. 2000) suggest that phosphorus can accumulate in the soils of ancient settlements as a result of food preparation and the utilization of waste products. The different phosphorus compounds in soils are stable to oxidation, reduction, leaching, and

dissolution (Lima da Costa & Kern 1999; Wells 2004). According to several scholars (Proudfoot 1976; Stevenson 1986), newly mineralized inorganic phosphorus, while generally retained in the soil, is subject to some vertical translocation in the soil due to factors directly affecting adsorption, such as pH, cation exchange capacity, and cation availability. The distribution of inorganic phosphate is therefore determined by the chemistry and adsorption kinetics of the soil throughout the period of decomposition and precipitation (Schlezinger & Howes 2000). Phosphorus is the main component of human and animal bones, a component of living tissues (in the form of nucleic acids, phospholipids, nucleotides, and so on), and a component of everyday products (such as wood, plant, or meat products) (Sanchez 2007). The phosphorus concentration in the soil increases depending on the supply of different organic materials, such as plants and animals, that are used by people. The phosphorus content in cultural layers is an indicator of the intensity of human occupation in the area. The concentration of organic materials containing phosphorus, accumulated in the process of human activity, is proportional to the time of human occupation and the growth of the population (Marwick 2005).

High concentrations of potassium (K) and sodium (Na) can be connected with the presence of fire ash in the areas of fireplaces (Middleton & Price 1996). In soils, potassium (K), sodium (Na), and rubidium (Rb) are the main components of feldspar and plagioclase minerals. Rubidium (Rb) could be taken up by plants as a substitute for potassium (K), which it chemically resembles. In this case, rubidium behaves like potassium. We can also use rubidium as an ash indicator for some types of deposits. We can therefore consider these elements as anthropogenic components if we have any other evidence for human activity at the archaeological site, such as anomalous concentrations in comparison with the background or a correlation between high concentrations and any remains of charcoal or fire ashes.

Increased concentrations of iron (Fe) and mercury (Hg) at settlements can be explained by the use of different natural pigments in rituals. The combination of elements such as Fe, Mn, Zn, and Cu can indicate areas of waste disposal, burials, cesspools, or rubbish after feasts (Wells et al. 2000). The chemical composition of soils in settlement areas can therefore provide information about different features of functional zones at settlements. According to most researchers, soils have the ability to withhold and absorb chemical elements resulting from anthropogenic activity during many years. At archaeological sites, these materials usually consist of household waste, bones, metal slag, ashes, dung, and the remains of burials and cremations. Investigations in the past few years (Wilson et al. 2005) showed that using just one chemical element or its compound to determine features of archaeological objects or functional zones at settlements is not always correct because the results obtained could be explained by several archaeological and natural contexts. The accumulation of different chemical components depends on the different natural factors of sedimentation and the further diagenetic transformations of sediments, the duration and intensity of human occupation at the site, and other factors. The method of "multi-element" analysis (Lima da Costa & Kern 1999; Parnell & Terry 2002; Wilson et al. 2005), which has been applied relatively recently, allows us to establish a complex of several chemical components and to consider their connection with different functional zones.

The first results in the development and application of phosphate analysis in the territory of the USSR were obtained in the 1950s. This method was applied to search for ancient settlements in Estonia and Latvia (Velleste, 1952; Shtobe 1959). In the beginning of the 1960s, phosphate analysis was used to search for Neolithic and Bronze Age sites in north-western Russia (Miklyaev & Gerasimova 1968). At present, phosphate analysis is extensively used to examine and search for archaeological sites in the territory of Russia (Anderson et al. 2009). In contrast, the multi-element approach is not commonly applied in Russian archaeology. Only a few sites were investigated with this method (Kulkova et al. 2012). These investigations are therefore very important for Russian archaeology and the further development of this approach is significant. At present, the method used in Russia consists of sampling a square of the site and applying multi-element analysis with data processing by mathematical statistics in order to reconstruct functional zones at archaeological sites (Kulkova 2012). The application of statistical analysis methods allows dividing the complex of chemical elements analyzed in anthropogenic soils into several groups.

The associations of chemical components in groups with close correlation bonds testify that these elements were formed in the same geochemical environments. We can determine the groups of chemical elements that are connected with different types of anthropogenic activity. Together with the method of multielement analysis, the indicator ratios of chemical elements can be applied to the reconstruction of functional zones. These indicator ratios of chemical elements reflect the degree of enrichment of anthropogenic elements in comparison to their background concentrations in the soils of the settlement and outside of it (Oonk 2009). The geochemical methods can be used at multilayer sites to analyze cultural layers in cases where the living and household structures have been destroyed. This method provides additional information for interpreting different areas of the site, such as pits, the remains of constructions, or zones around fireplaces. The geochemical analysis of soils and deposits was carried out at two archaeological sites located in the Neva river basin in order to reconstruct their functional zones.

The archaeological sites under study in the Neva river basin

The most thoroughly studied archaeological sites at which the interdisciplinary investigations have been carried out are Okhta 1 and Podolije 1 (Figure 1) (Gusentsova & Sorokin 2011; Kulkova et al. 2014; Gusentsova et al. 2014). It should be noted that the main period of their occupation was at the same time during the Late Neolithic and Early Bronze Age. The method of geochemical investigations was applied to the study of cultural layers connected with a date of approximately 3300 cal BC (Gusentsova et al. 2014).

The Neolithic-Early Iron Age site of Okhta 1

The Okhta 1 site is located in the center of St Petersburg near the confluence of the Neva and Okhta Rivers. The cultural remains of the ancient site were excavated in an area larger than 10,000 m². Excavations in the central and



Figure 1. Locations of the Okhta 1 and Podolije 1 sites.

southern parts of the study area encompassed about 6,700 m² (excavations in 2008–2009). The cultural layers of the prehistoric settlements are situated under alluvial sandy sediments with a thickness of 1-1.5 m, lying under the buried soil of the Middle Ages. So far, this is the only site dating to the Neolithic-Early Iron Age known from St Petersburg. The collection of archaeological finds totals about 1,200 objects, including pottery, stone tools, wooden carvings, and amber jewellery. The remains of wood constructions were discovered for the first time in the St Petersburg region. These artefacts are mostly wood piles, strips, and rails. Due to the number of archaeological finds and the preservation of organic finds, the Okhta 1 site is a unique and rare object of cultural heritage in the territory of the Baltic Sea and Northern Europe. The results of interdisciplinary investigations provided us with a significant database for reconstructing the palaeogeographical conditions, the processes of cultural layer formation, and the development of cultural-historical stages in the region. These results have been published elsewhere (Sorokin et al. 2009; Kulkova et al.

2012; Gusentsova & Sorokin 2011, Kulkova et al. 2014a, Kulkova et al. 2014b).

Neolithic-Early Bronze Age site of Podolije 1 The Podolije 1 site is located on the southern shore of Lake Ladoga at an absolute height of 11.8-12.1 m. During the period of settlement, the site was situated in the coastal zone of Lake Ladoga. When the Lake Ladoga transgression occurred, the site was flooded and people abandoned it. Two different cultural layers can be identified at this site. The upper cultural layer consists of yellow-grey sand with a thickness of 0.2-0.8 m. The lower cultural horizon consists of peat interlaid with yellow sand (the thickness of the cultural layer is 0.8 –1.5 m). The peat was formed in conditions of shallow water in a swamped basin. Archaeological finds are represented by an assemblage of different types of pottery, a collection of stone and bone tools for hunting and fishing, amber adornments, and wooden remains of fishing constructions. According to the radiocarbon date, the site dates from the 5th to the 3rd millennium BC (Gusentsova et al. 2014).

Materials and methods

At the site of Ohkta 1, the deposits were sampled from a square of the cultural horizon consisting of yellow sand located on a grid of 7.8 x 6 m in excavation № 7/2–1 (Figure 2). The soil samples were taken at intervals of 0.6 m on the coordinate grid. The coordinates of the primary point of sampling were registered by GPS. The 154 samples were sampled and analyzed. According to the archaeological finds, this zone can be interpreted as a burial place. Finds from this location include a human tooth, several amber adornments, traces of red ochre, and a KULKOVA, GUSENTSOVA, NESTEROVA & NESTEROV



Figure 2. The sampling location in excavation № 7/2-1 of the Okhta 1 site.

stone construction. Not very many archaeological finds were found inside the burial area. There are several spots of red-brown sandy loam including charcoal particles and several small fragments of ferruginous bones. The human tooth was found among the bones. In the northern part of one of the red spots, there was a circle built of several big boulders. The necklace, which was made of 13 amber oval pendants resembling buttons with V-holes, was found close to the red spot. Two other pendants were found close to other red spots. The oval stone construction can be seen on the excavation plan (Figure 2). The geochemical method was applied to check this location.

Sampling at the Podolije 1 site was conducted on a square of excavation 2 (sq. 4-11/X-III) on the surface of the yellow-grey sand at intervals of 0.5 m on the coordinate grid (Figure 3). Altogether 76 samples were analyzed from this area. In this part of the excavation, the narrow terrace of an ancient channel had been revealed on the basis of geomorphological data. In the shore zone of this channel, earth structures with fragments of pottery, stone tools, animal bones, and charcoal were found. The three background samples were taken from the same layers outside of cultural horizons.

The chemical composition of the deposits from the Okhta 1 and Podolije 1 sites was determined by XRF analysis by means of the vacuum wavelength dispersive X-ray fluorescent WD-XRF scanning spectrometer "Spectroscan Makc-GV" in the Lab of Geochemistry of the Environment of Herzen State University. This is a desktop WD-XRF spectrometer controlled by an external computer. The measuring system of the spectrometer is in the vacuum chamber, while the samples are at



Figure 3. The sampling location in the excavation of the Podolije 1 site.

the ambient pressure, so no He is required for the sample chamber and all samples (including liquid and powder) may be studied with no special measures taken. Previously, the deposit samples were dried in an oven for 24 hours at a temperature of 105 °C. After this procedure, the removal of large particles (generally particles greater than 2 mm in diameter), and sample homogenization to powder in an agate mortar, about 1 gram of powdered sample was fired in an oven for the burning of organics at a temperature of about 550 °C for 40 minutes. The samples were pressed in tablets used for measurement in the XRF spectroscan.

The Surfer Mapping Software (Version 8.0) was applied to map the distribution of geochemical indicators of anthropogenic activity in the studied area.

The results of the geochemical investigations

The Okhta 1 site

Based on the geochemical analysis, there are anomalies in the concentrations of several chemical elements connected with anthropogenic activity in the cultural deposits from the sample of excavation Nº 7/2-1 at the site of Okhta 1 in comparison with their background concentrations (Nesterov et al. 2011, Kulkova et al. 2014b). The distribution maps of phosphorus $P_2O_5(\%)$, anthropogenic calcium $CaO_{antr} = CaO/(CaO+Na_2O)$ (%), and iron $Fe_2O_3(\%)$ in deposits in the sampled square of cultural layer are presented in Figures 4 and 5. The indicator CaO_{antr} was calculated as the ratio between CaO_{tot} and the sum of CaO_{tot} and Na_2O . In this case, we separated anthropogenic calcium from lithogenic calcium, which is found in the mineral composition of deposits. The concentrations of other anthropogenic elements in the deposits of this site are on the background level. These elements (MnO, K₂O, and Rb) were therefore not used for reconstructions (Figure 6).

The Podolije 1 site

At the Podolije 1 site, there are important indicators that help to understand the features of the ancient terrace microrelief. The map of artefact distributions is presented in Figure 7. The variations in the concentrations of the main chemical components of sand deposits, such as aluminium (Al₂O₃,%) and silica (SiO_{2.}%), were used for this purpose. The distribution maps of these components reflect the changes in the ancient microrelief at the excavation site (Figures 8 and 9). The high concentration of phosphorus (P₂O₅, %) (Figure 10) is registered in the big depression in the ancient channel. In the deposits of this depression, high concentrations of manganese (Mn), barium (Ba), and iron (Fe) were registered (Figure 11). In order to separate the component fraction of anthropogenic origin from the lithogenic fraction, the ratio of concentrations of total chemical elements in the deposits was used. The anthropogenic calcium CaO_{antr}(%) was calculated as $CaO_{antr} = CaO/(CaO+Na_2O)(\%)$ and its distribution is shown in Figure 12. In the case of these sites, sodium (Na₂O) has a totally lithogenic character. The map of anomalies in anthropogenic strontium $(Sr_{antr}) = Sr/(Sr+Na_2O)(\%)$ is presented in Figure 13. Calcium is replaced by strontium in the carbonate apatite of bones. The distribution of concentrations of $K_2O_{antr}=K_2O/K_2O+Na_2O)(\%)$ and Rb_{antr}=Rb/(Rb+Na₂O)(%) are shown in Figures 14 and 15. In the sediments of this site, potassium and rubidium have a strong correlation with each other and are connected with the ash component of this site.

Discussion

At the Okhta 1 site, the geochemical anthropogenic indicators connected with the accumulation of bones and tissues (phosphorus P_2O_5 (%) and calcium CaO/(CaO+Na₂O)(%)) have anomalies in the area where a human tooth was found. In this location, an anomaly in the concentration of iron $(Fe_2O_3(\%))$ is registered as well. The highest iron concentrations are connected with the areas of red spots. The mineral composition of these sediments consists of ochre minerals. Anomalies in the iron concentration could therefore indicate ritual activity in this place. In areas where there were anomalies in the combination of these elements, different archaeological artefacts, such as the tooth, amber adornments, ochre, and a stone construction were found. The locations of these finds are connected with the inside area of the contours of the main element indicators. All this evidence gives reason to believe that this place was used for burial in the 4th millennium BC.

The geochemical mapping of the whole excavation square revealed that the areas of anthropogenic indicators have maximums in the area connected with burial. The character of geochemical indicator distributions and the character of sedimentation (Kulkova et al. 2014b) reveal that the burial had taken place in the shore zone of the shallow Littorina Sea and that it was partly rewashed during the transgressive stage of the basin. The geochemical data on anthropogenic activity indicate maximum concentrations in the zone with artefacts, whereas the concentrations in deposits of other parts of the excavation are extremely low. The relatively high concentrations of anthropogenic elements in comparison to the background concentrations, as well as the presence of burial artefacts, serve as evidence



Figure 4. The distribution of phosphorus (%) and anthropogenic calcium (%) in the deposits of excavation 7/2 (Okhta 1 site).



Figure 5. The distribution of iron (%) in the deposits of excavation 7/2 (Okhta 1 site).

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Figure 6. The distribution of potassium (%), manganese (%), and rubidium (ppm) in the deposits of excavation 7/2 (Okhta 1 site).



Figure 7. Artefact distribution of the Podolije 1 site.

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Figure 8. The microrelief map of alumina distribution (%) in the deposits of the Podolije 1 site.



Figure 9. The microrelief map of silica distribution (%) in the deposits of the Podolije 1 site.



Figure 10. The distribution of phosphorus in the deposits of the Podolije 1 site.

against any significant rewashing of deposits. In the case of the redeposition and transposition of sediments together with artefacts, anomalies in the concentrations of anthropogenic indicators would not be revealed and the deposits would have background-level contents of anthropogenic elements and indicator ratios. The artefacts were found *in situ* in this cultural layer and the geochemical characteristics of the cultural deposits confirm their positions. The data obtained show that the anomalies in the distribution of anthropogenic elements have a good correlation with the remains of the ancient stone construction and the artefact finds (Figures 4 and 5).

The high concentrations of aluminium at the Podolije site were revealed at elevations of the relief that consists of clay loam. Silica is the main component of sand, and its accumulation and high concentrations mark the channel in relief. This indicator marks a depression formed during the ancient channel development. The largest amount of artefacts was found on the sand shore of the channel. There is a good correlation between the altitude of the microrelief, the concentrations of clay and sand sediments, and the alumina and silica distributions. The 3D reconstruction of the microrelief of this place and the correlation with the aluminium distribution is shown in Figure 16.

The distribution maps of anthropogenic components show the anomalies connected with the different functional types of human activity that occurred on the shore of this channel. A high concentration of phosphorus (P2O5, %) (Figure 10) is registered in the big depression in the ancient channel located near the edge of the terrace. In the deposits of the terrace, high concentrations of manganese (Mn), barium (Ba), and iron (Fe) were registered (Figure 11). Their anomalies can be connected with the remains of decaying organics on the slope of the hill. Different materials from the top zone probably accumulated in this part of the terrace as a result of erosion processes.

Anthropogenic calcium is a good marker for several zones located on the shore of the channel. In these zones, various archaeological finds such as stone tools, ceramic sherds, and bones were found. These zones can be interpreted as places in which ancient people could prepare animals and fish (Figure 12). High concentrations of anthropogenic strontium were also registered in these places. Elements such as CaO_{antr} , Sr_{antr} , and P_2O_5 are the main components of the remains of animal and fish bones. The reconstruction of the locations of fish and animal preparation zones is shown in Figure 16.

There are several places in which fireplaces were located. Charcoal and ash were found in these places, and they are characterized by maximally high concentrations of anthropogenic potassium and rubidium (Figures 14 and 15). Several fireplace zones coincide with places of fish and animal preparation, which are marked by CaO_{antr} and Sr_{antr}. The increased concentration of these elements was determined also in some parts of the channel (Figure 16).

The data obtained therefore allows us to determine several zones in the shore part of the terrace. These zones can be interpreted as temporary places used for fishing and animal preparation. These places are situated near hearths that had been used for cooking on the edge of the terrace. In the pit located at the edge of the terrace, most of the waste material was accumulated in processes of sediment rewashing and the erosion of elevated loam hills.

Conclusion

Two different types of functional zones were considered at the Ohkta 1 and Podolije 1 sites occupied by people in ca. 3300 cal BC. At the Okhta 1 site, the data provided by geochemical mapping and distributions of artefacts allows determining the burial place. The burial is characterized by a maximum combination of chemical anthropogenic components such as P_2O_{5antr} , Ca O_{antr} , and Fe $_2O_3$. Another type of distribution of chemical elements was considered at the Podolije 1 site. There were seasonal sites with fireplaces and zones for fishing and animal preparation on the shore terrace of the channel. In the deposits of this site, a combination of anthropogenic elements such as K2Oantr, Rbantr, CaO_{antr}, and Sr_{antr} was defined. The depressions on the shore of the channel located at the edge of the terrace are characterized by anomalies of a set of chemical elements, namely K₂O_{antr}, Rb_{antr}, CaO_{antr}, Sr_{antr}, Mn_{antr}, Ba_{antr}, Fe, and P₂O_{5antr}, which can be interpreted as refuse from the hills. The geochemical indicator combinations from the same lithological context at the Okhta 1 and Podolije 1 sites have different meanings. The burial zone is characterized by anomalies in bioindicators (P₂O_{5antr} and CaO_{antr}), which are the main components of bones and tissues, as well as iron (Fe_2O_3), which was a component of the ochre used during rituals. A geochemical complex including K_2O_{antr} , Rb_{antr} , CaO_{antr} , and $Sr_{_{antr}}$ characterizes fireplaces ($K_{_2}O_{_{antr}}$ and $Rb_{_{antr}}$ are microelements of coals and ash) and zones of fish and animal preparation (CaO_{antr} and Sr_{antr} are elements of bones). In the depressions on



Mn (%)



Fetot(%)

Figure 11. The distribution of manganese (%) barium (%), and iron (%) in the deposits of the Podolije 1 site.

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0.085 0.08 0.075 0.07 0.085

0.06 0.055 0.05

0.045

0.04

0.035 0.03 0.025 0.02 0.015

0.01

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Figure 12. The distribution of ${\rm CaO}_{_{\rm antr}}$ (%) in the deposits of the Podolije 1 site.



Figure 13. The distribution of Sr_{antr} (%) in the deposits of the Podolije 1 site.

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Figure 14. The distribution of K2Oantr (%) in the deposits of the Podolije 1 site.



Figure 15. The distribution of Rbantr (%) in the deposits of the Podolije 1 site.



Figure16. The 3D reconstruction of the locations of fish and animal preparation zones (a) and the microrelief on the basis of Al2O3 distribution (b).

the channel shore, material from the hills was found and registered with the help of complex of geochemical indicators: K_2O_{antr} and Rb_{antr} are elements of ash, P_2O_{5antr} , CaO_{antr} , and Sr_{antr} are elements of bones, and Mn_{antr} , Ba_{antr} , and Fe are elements of decayed organics.

The geochemistry of the distribution of main sediment-forming elements, such as alumina (Al_2O_3) and silica (SiO_2) , is important for the determination of the ancient microrelief at the site.

The research of archaeological areas by means of geochemical indicator mapping provides an opportunity to assess the functional zones of archaeological sites in more detail. This data allows us to characterize the different types of functional zones and to reconstruct human life at these settlements. These investigations were carried out for the first time in this region, and this method should be applied more widely in Russian archaeology. The development of the multi-element method for the reconstruction of functional zones at sites dating from the Neolithic to the Early Iron Age is important for understanding the activities of ancient people in the territory of Russia and other regions.

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