THE METODS OF RESEARCH OF BURIED SOILS UNDER ARCHAELOGICAL SITES

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ABSTRACT

One of the ways to study the evolution of natural environment, in particular, the soil cover is a comprehensive study of soils buried under archaeological sites. Comparative analysis of the properties of paleosols buried in different historical epochs allows us to examine in detail the spatial and temporal dynamics of the natural environment and its components in time. The use of such comparative analysis is possible because the burial of soil under embankments of defensive ramparts, burial mounds and cultural layers of ancient settlements isolates the soil from the influence of external environment, providing it with a kind of "conservation." Besides, the soils are capable of preserving a whole range of non-pedogenic traits that can be used for a more detailed reconstruction of the features of the natural environment at the time of the soil burial. So, in the soil profile, spores, and pollen of plants, phytoliths, faunal residues, etc. remain. The use of soil methods in the study of archaeological sites in Russia began more than a century ago, but the development of the scientific foundations of a new direction in soil science, called archaeological soil science, began in the 1970s and 1980s. To study the buried soils under archaeological sites, it is necessary to use a set of methods, since often a method alone has its limitations for obtaining reliable results.

Keywords: buried soils, archaeological sites, Holocene, soil chronosequence, methods.

INTRODUCTION

The study of the conditions of the natural environment of past eras is one of the priorities of natural sciences. Objects, which can save paleoinformation, include soils buried under different archaeological structures: the burial mounds, defensive ramparts, cultural layers. Buried soils in a state of conservation, retain a large amount of information about the past stages of soil formation. The age of these soils is limited to the second half of the Holocene, because the steppe Eneolithic tribes of the South of the East European plain applied a new type of burial rite about 6000 years ago: construction of a mound above the burial pit. The tradition of constructing mounds for the burial were preserved among the tribes from the Bronze (III–II thousand BC) to the Early Iron (I thousand BC – IV century AD) Ages and the early and the developed phases of the Middle Ages (V–XIV centuries AD).

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The study of the paleosols under the different age mounds of the Eneolithic, Bronze, Early Iron and Middle Ages allows both to obtain the new notions regarding the evolution of the natural environment in those time periods and to analyze the dynamics of the settlement of ancient people. In recent decades, paleo information is obtained using a number of methods, which, in addition to the traditional morphological, chemical and physico-chemical analyses, include radiocarbon, isotopic, spore-pollen, microbiological, molecular genetic, phytolithic, micromorphological, geochemical and other analyses.

The use of a complex of methods in the study of buried soils allows us to clarify a number of both fundamental and applied tasks: 1) Holocene trends in the evolution of soils and soil cover, 2) the centennial dynamics of changes in soil properties due to spatial and temporal variability of soil formation factors, 3) shifts of landscape boundaries and associated processes of migration, settling, economic activities of the ancient and medieval population.

The study of buried soils under archaeological sites has some specific features since after its burial the soil is often transformed by diagenesis [1, 2, 3]. Accordingly, it is necessary to take into account the stable features of soils, which include a vertical sequence of genetic horizons, mineralogical and granulometric composition, the structure of illuvial horizons, poorly soluble segregated neoformations, and many features of a microstructure of soils. The content of humus due to mineralization of organic matter with time, the structure of eluvial horizons, the density of soil substrate, the composition and distribution of water-soluble salts along the profile, and the soil-absorbing complex are among the diagenetic features. The degree of diagenetic changes in a soil profile depends on the thickness and composition of the mound which covers the buried soil and the depth of groundwater table. Also, the factors affecting the properties of buried soils can be the burrowing activity of the fauna and the pressure of the earth walls. The density of the humus horizon buried beneath the embankments of soils is higher by 10-15% than that of the surface soils [1].

Morphological method.

In spite of the emergence of new methods, one of the most important approaches in paleopedology is a careful study of morphological properties and features of buried soils with the identification of relict properties and diagenetic features. Traditional morphological survey of soils in recent years is complemented by mesomorphology, showing the details of various soil characteristics. So, with sufficiently high zooming, the time sequence of elementary soil processes becomes visible: carbonate formation of carbonates over clay films, traces of gleying, etc.

Methods for determining soil age.

The most reliable method of dating buried rocks is radiocarbon analysis, which allows to obtain dates in the interval from hundreds of years to 40-60 thousand years. Radiocarbon analysis is based on the determination of the age of carbon-containing materials, by measuring their radioactivity using the isotope 14C with time of half-life equal to 5730 ± 40 years [4]. At present, there are two methods of radiocarbon dating: 1) the method of liquid scintillation counting (LSC); 2) method of accelerating mass spectrometry (AMS).

To solve the problems of archaeological chronology, the objects for radiocarbon dating are soil carbonates, soil humus, charcoal, bones and other materials of organic origin from archaeological burials. During the application two dates are distinguished: *radiocarbon* age and *calibrated* age marked by the symbol "cal" [5] which is corrected for changes in the concentrations of 14C occurring in atmospheric carbon (CO, CO_2) over time. The results of radiocarbon analysis may be distorted if a sample was heavily contaminated with carbonaceous materials of a later period.

Interpretation of radiocarbon dates is especially problematic when soil organic material is analysed since soils are an "open-closed system" for carbon exchange: most humic substances are mineralized and renewed in the process of soil formation. Humic substances are the most ancient and stable humic acids, but they are subject to renewal. Consequently, the dates obtained for organic matter speak instead about the rate of carbon metabolism, and not the age of the soils.

Paleobotanical method.

The best known paleobotanical methods for the reconstruction of natural conditions existing in past epochs of soil formation are spore-pollen and phytolite analysis. The first one is based on the fact that plants during the flowering period produce a huge amount of pollen spores of an individual shape for each kind of vegetation. When deposited on the soil surface, they create a spore-pollen spectrum reflecting the vegetative composition of the region [6]. The preservation of these particles is hundreds of thousands and millions of years. As a result, it becomes possible to obtain data on the species composition of the vegetation cover for a certain historical period and also reconstruct the climatic conditions.

The preservation of pollen grains might be insignificant in cultural layers and buried soils, and individual plant families may disappear from the spectrum, which distorts the final interpretation of the results. Consequently, the data of spore-pollen analysis of soils should be treated with caution and, if possible, compared with the results of other paleobotanical methods.

Phytolith method.

Phytolith analysis is based on the fact that practically all plant organisms, including algae and higher plants, uptake dissolved silica. As a result of the silica accumulation in various organs of the plant (in intercellular spaces, inside cells or on the surface of leaves), biominerals made of amorphous silica, called phytoliths, are formed. After the plant die, phytoliths enter the soil, accumulating in the upper humus horizon and remaining there for thousands of years [7]. Since the preservation of phytoliths in buried soils is high enough, they can serve as indicators of particular plant communities of the past.

Archaeological method.

The study of material remains of past archaeological cultures forms the basis of archaeological method. The material remains include: 1) objects created or processed by people - tools, ornaments, clothing, clay vessels, waste products, slag pieces, etc.; 2) structures created by people - ground constructions for storing products or for rubbish pits, traces from pillars, the dark contour of rotted or burned wooden logs, a hearth filled with charcoal and ash; 3) biological remains - untreated bones, snail shells, plant pollen, charred grains and wood.

Archaeological method is a tool for determining paleosols' age. Archaeologists have created regional archaeological calendars when studying the forms of ancient objects, the ornamentation of vessels, and the stratigraphy of cultural layers. Regional periodization systems are corrected by radiocarbon analysis, which increases the reliability of the received dates. The accuracy of archaeological dating for the last 4-5 thousand years is 250-300 years. Within the territories with a high population density of the archaeological sites studied, the accuracy of archaeological dating is even less (25-50 years).

Various chrono-reference characteristics also help to determine the soil events over timescale. For example, in 1630 BC there was a catastrophic eruption of the Tera volcano on the island of Santorini in the Aegean Sea, accompanied by sharp, prolonged cooling of the climate in the northern hemisphere. This event is a historical reference if layers of volcanic ash are revealed on the surface of buried soils.

Historical method.

The historical method of dating is indirect and based on the study of soil processes from written historical sources, maps, eyewitness documents. This method covers the events of the last three to four centuries. The use of historical sources allows to reconstruct the water supply of the territory, fluctuations in the sea level and lakes, also to fix the changes in river network and the direction of river flows, etc.

Elemental analysis.

Elemental analysis of paleosols performed with help of modern multi-elemental analytical techniques (XRF, ICP-OES/AES, ICP-MS) is used to solve several types problems. The first problem concerns the evaluation of the lithological factor: the provenance of the deposits on which the soils were formed and also vertical differentiation of soils influenced by lithological discontinuities. One of the main approaches to do this is the calculation of the molar ratios of relatively immobile elements [8] (Ti/Al, Zr/Y, Ti/Zr, Zr/Nb, etc.). The criterion of lithological homogeneity of sediments, according to many authors, is a small vertical variability of these parameters and small differences between their unaltered parent materials.

Another important area of geochemical analysis in paleosol studies is quantification of the individual soil processes that can be detected as transformation of the elemental composition of the initial parent material. An objective assessment of profile differentiation is important in establishing the genesis of soils, but requires consideration of the specific soil features: different chemical composition of soils needs different approaches for the analysis. For example, geochemical proxies developed for the analysis of non-calcareous soils cannot be used to quantify the processes in soils containing readily soluble salts and carbonates. One of the common methods of quantitative characterization of individual pedogenic processes is the calculation of indicator molar ratios [8] of major and trace elements, such as $Sa = SiO_2 / Al_2O_3$, $Saf = SiO_2/(Al_2O_3 + Fe_2O_3)$, $CIW = Al_2O_3/(Al_2O_3 + CaO + Na_2O) \times 100$, Ba/Sr, Rb/Sr, etc. Calculation of the coefficients for the clay fraction additionally characterizes its mineralogical composition, therefore make the interpretation of proxies more reliable.

Another problem solved with the help of soil elemental analysis is the detection of specific traces of anthropogenic activity that are morphologically invisible [9]. The revealing geochemical signatures and markers of anthropogenic impact can be carried out by comparing the contents of elements in the analogous horizons of the buried paleosols and non-polluted surface soils [10] or through application of statistical methods. The revealed associations of elements and their concentrations can give important information about the sources, nature and intensity of anthropogenic impact, and help in reconstructing the lifestyle of the people in the past eras.

Micromorphological method.

With the help of micromorphological studies of soils in thin sections, diagnostics of the basic patterns of microstructure under the polarization microscope (composition and structure of aggregates, pore space, degree of weathering or stability of minerals, evaluation of the mobility of finely divided mineral, humic and organic substances, the identification of various soil neoformations) which can clarify the genesis and evolution of surface and buried soil. A specific feature of micromorphological method is the ability to trace a unique combination of elements of microstructure which is specific for different soil-climatic conditions (arid, humid, etc.) and/or for various types of anthropogenic influences.

The principal task in the research of paleosols using the micromorphological method is the extraction of pedogenic morphological records from soil memory at microscopic level. The core of soil memory lies in the fact that the in situ interactions of soil formation processes are recorded in the solid phase of soil multiphase system and are reflected at different levels of soil organization with varying degrees of resolution [11]. Microscopic level of soil mass organization allows reconstructing the paleoenvironmental conditions before the burial of soils [12].

The use of the micromorphological method in the study of paleosols is based on the principle of actualism when elements of microstructure of buried soils are compared with the diagnostic features of surface soil. In the opinion of [13], in many cases, this approach allows us to determine soils up to the highest (type) level of classification. According to the stability both in time and during pedomorphism, micro-signs are divided into three groups: 1) stable - the microstructure of the matrix, the elementary micro composition - the ratio of coarse and fine particles (K / T), the structure of aggregates, the shape of pores, the type of microstructure of the finely divided material (plasma), clay cutans; 2) weakly stable - humus; 3) unstable - saline neoformations [13]. Modern research allows us to say that such a division of micro-signs by the degree of stability is characteristic of soils that are formed (or have already been formed) under humid conditions. For arid conditions of pedogenesis within the group of salt neoformations, gypsum is the most informative for carrying out paleoreconstructions, since it is relatively resistant to the time factor and can diagnose the impact of hydrogenous paleoconditions during pedogenesis [14,15].

Micromorphological analysis makes it possible to separate anthropogenic and natural features, especially in the presence of such inclusions as fragments of bones, plant detritus, phytolites, products of the destruction of adobe construction materials, clusters of calcite spherulites, imported with manure, textiles, carbonaceous material, ashes. Microremains of materials of plant and animal origin may well occur in natural soils and sediments that have not experienced anthropogenic impact. However, the presence of such inclusions in large quantities in the archaeological context, in pedolithic sediments (cultural layers), as well as their uneven distribution across stratigraphic units of the cultural layer, can be indicative of precisely the anthropogenic origin of these

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traits. In addition, one can see the signs associated with the impact of fire or high temperatures: wood and grassy coals, ash, accumulation of pyrogenic carbonates, calcined aggregates of silicate material, microfragments of ceramics, accumulations of vitrified phytolites, as well as phosphate, phosphate-humus, phosphate-ferruginous neoformations formed as a result of the transformation of the compounds of phosphorusderived from organic matter of animal origin. Thus, micromorphological studies are an essential and necessary complementary method in the study of paleosols.

Method for determining the soil genome.

With molecular biological methods, such as extraction of total DNA from the soil, quantitative PCR (qPCR) and high-throughput sequencing (NGS), it is possible to investigate soil microbiome – all microbial genetic material in the system. The object of analysis in this case is the cumulative DNA of living microorganisms, dormant forms, dead cells, as well as extracellular DNA adsorbed on soil particles. Considering high diversity and variability of the soil microbiome, analysis of the microbial DNA in buried soils can be a sensitive indicator of soil burial conditions and organic matter composition and quantity, and can carry possible information about conditions and vegetation before burial as well. One of the main problems in interpretation the results of these methods is the inability to distinguish the contribution of the "modern" DNA of buried soil microbial communities and the "ancient" DNA resting from the time before burial. Thus, it is difficult to distinguish that part of microbiome can serve as an indicator of soil diagenesis changes and one that can be a marker of paleoclimatic reconstructions. Possible solutions to this problem may be DNA fractionation by isotopic composition or paired analysis of soil DNA and RNA.

Carbonate profile of soils.

Since the most widespread parent material in the forest-steppe and steppe zones of the East European Plain are loess and loess-like loams characterized by a high content of carbonates, one of the informative indicators of revealing trends of soil formation is identification of horizons with various accumulative forms of carbonate neoformations as well as the features of their depth distribution. Numerous studies of paleosols show that the depth distribution of carbonates indicates a change in water regime types. The main problem that arise in the study of the soil carbonate profile is the possible translocation of carbonates from the mound overlying the buried soil. In this case, careful morphological studies of the soil substrate at the boundary of the soil of the embankment and paleosols are necessary. If the embankment is sufficiently thick (about 1 meter or more) and has a clayey texture, the supply of carbonates to the profile of a buried soil does not practically occur. Besides, it is problematic to study the carbonate profile of buried soils in archaeological sites located in hydromorphic and semi-hydromorphic positions of the relief with a close groundwater table. In this case, the carbonate profiles are superimposed and the initial version of the profile at the time of the soil burial is practically impossible to establish. The presence of pyrogenic carbonates complicates the investigation of the carbonate profile. Such pyrogenic carbonates originated usually as a result of the performance of funeral rite often accompanied by the fires, as evidenced by the numerous coal remains.

CONCLUSION

Reconstruction of environmental conditions in the past eras can be carried out by examining buried soils under archaeological sites. Every study object needs application of its own set of methods.

1. For all objects it is necessary to use cross-cutting methods: morphological analysis carried out at various levels, spore-pollen and phytolith analyses, as well as the geochemical analysis of the soil substrate.

2. For the soils of the forest and forest-steppe zones, in addition to cross-cutting methods, it is necessary to evaluate the intensity of textural differentiation. This can be done analytically using the profile differentiation coefficients, and morphologically (revealing the cutan of illuviation, the degree grains's bleaching). The content and composition of humus in buried soils, the presence of paleokrotovinas, the depth of occurrence of carbonate segregated neoformations help to determine the change in landscape conditions.

3. For steppe soils characterized by a non-percolating type of water regime, in addition to the methods that determine the thickness of humus horizons, the content and reserves of humus, the depth of occurrence and the forms of carbonate neoformations, it is necessary to define the contents of gypsum and readily soluble salts, which help to reveal desalinization and salinization trends.

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REFERENCES

[1] Evolution of soils and soil cover. Theory, diversity of natural evolution and anthropogenic transformations of soils, Editors V.N. Kudeyarov, I.V. Ivanov. Moscow, GEOS, 2015, 925 p. *(In Russian);*

[2] Ivanov I.V., Evolution of soils of the steppe zone during the Holocene, Moscow, 1992, 143 p. *(In Russian);*

[3] Puzanova T., Makeev A., Khokhlova O., Aseeva E., Kurbanova F. Holocene signals of climate dynamics in the profiles of soils buried under archaeological objects in the central part of the East European plan. SGEM 2017 Conference Proceedings, Vol. 17, Issue 32, 2017, pp. 273-281, DOI: 10.5593/sgem2017/32/S13.036;

[4] Godwin H. Half-life of Radiocarbon, Nature, 195, 1962, 984 p.;

[5] Reimer PJ, Bard E, Bayliss A, Beck JW, Blackwell PG, Bronk Ramsey C, Buck CE, Cheng H, Edwards RL, Friedrich M, Grootes PM, Guilderson TP, Haflidason H, Hajdas I, Hatté C, Heaton TJ, Hogg AG, Hughen KA, Kaiser KF, Kromer B, Manning SW, Niu M, Reimer RW, Richards DA, Scott EM, Southon JR, Turney CSM, van der Plicht J. IntCal13 and MARINE13 radiocarbon age calibration curves 0-50000 years calBP. Radiocarbon, 4, USA, 2013, pp. 1869-1887;

[6] Grischuk V.P. Reconstruction of the scalar climatic indices with the floristic data assesment of its accuracy, Methody reconstruktsii paleoklimatov, Russia, 1985, pp. 20-28 *(In Russian);*

[7] Golyeva A. A. Biomorph analysis as a Component the Genetic and Morphological Study of Soil, Pochvovedenie, 1vol. 9, Russia, 1997, pp. 5-13 *(In Russian);*

[8] Sheldon N.D., Tabor N.J. Quantitative paleoenvironmental and paleoclimatic reconstruction using paleosols'. Earth-Science Reviews, Vol 95, no. 1-2, 2009, pp. 1-52;

[9] Canti M., Huisman D.J., 2015. Scientific advances in geoarchaeology during the last twenty years. J. of Archaeological Science 56, pp. 96-108;

[10] Aseeva E., Makeev A., Rusakov K., Sorokina K., Kurbnova F., 2017. The use of geochemical parameters to derive information on palaeo-environmental conditions from buried soils of an archeological site. SGEM2017 Conference Proceedings, Vol. 17, Issue 32, 2017, pp. 671-678, DOI: 10.5593/sgem2017/32/S13.087;

[11] Targulian V. O., "Soil memory: development, carriers, and spatial-temporal diversity," in Soil Memory: Soil as a Memory of the Biosphere-Geosphere-Anthroposphere Interactions (LKI, Moscow, 2008), 2008, pp. 24–54. *(In Russian);*

[12] Interpretation of micromorphological features of soils and regoliths / (Eds. G. Stoops, V. Marcelino and F. Mees), Elsevier, Amsterdam, 2010, 720p.;

[13] Morozova T.D. The development of the soil cover of Europe in the late Pleistocene, ed, A.A. Velichko, Russia, 1981, 281 p. *(In Russian);*

[14] Dobrovolskyi V.V. The manifestation of hypergenesis in quaternary sediments of the desert zone, *Gipergenez I kori vyvetrivania*, (Hyper genesis and Weathering Crusts: Selected Works), vol. 1, 2007, pp. 23-57 *(In Russian);*

[15] Lebedeva-Verba M.P. Micromorphological analysis and its possibilities in the study of paleosols. In «Paleopochvi, prironaya sreda I metoi ih diagnostiki» ed. Dobrovolskyi G.V., Dergacheva M.I., ch. 4, Russia, 1981, 281 p. *(In Russian)*