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Multidisciplinary Study of Burnt Deposits at Surungur, Fergana Valley, Southern Kyrgyzstan

Burnt deposits are an important source of information on ancient lifestyles, providing the possibility of reconstructing the size, intensity of use, and functions of fireplaces at prehistoric settlements, and to assess fuel sources. We outline the results of a multidisciplinary study of fireplaces and their contexts at Surungur—a stratified site in the Fergana Valley, in southern Kyrgyzstan. Sixteen samples from ash lenses and intermediate deposits were studied by rock-magnetism, gas chromatography mass spectrometry (GC-MS), and X-ray fluorescence (XRF). The rock-magnetic analysis suggests that the origin of all samples from ash lenses was anthropogenic. Types of fuel were reconstructed. At the initial stage (Early Holocene), the encompassing deposits likely resulted from short-term occupation, and fuel consisted of wood and grass/dung. In the Middle Holocene, occupation became more long-term, as evidenced by maximal heating temperatures and high concentration of fireplaces. During the Late Holocene, habitation intensity on the platform under the stone ledge remained the same, but heating was less intense. Wood and grass/dung were used as fuel at all stages, suggesting that wood was available in the region throughout the Holocene.

Keywords: *Fergana valley, archaeological site, fireplace, X-ray fluorescence (XRF), gas chromatography mass spectrometry (GC-MS), rock-magnetism.*

Introduction

Traces of fire use are an important archaeological source. Their analysis makes it possible to reconstruct specific aspects of everyday life and adaptive strategies used by the people of the past. The use of a multidisciplinary approach provides the opportunity to establish the features of hearths, such as the area of the fire-spot, center of the hearth, temperature threshold, and type of fuel used for keeping a fire, as well as to reveal thermally altered surfaces and artifacts (Nesterova, 2019). Physical and chemical methods are important components of geoarchaeological studies, proven to be highly effective and helpful (March, 1996).

The rock-magnetic method has been used in the studies of thermally altered objects from archaeological sites (Carrancho et al., 2009; Jrad et al., 2014). It is based on the idea that nonmagnetic or weakly magnetic minerals often transform into stronger magnetic phases in the course of mineralogical transformations resulting from heat exposure (Aldeias et al., 2016). Ashes are deposits with increased values of rock-magnetic parameters. Examining rock-magnetic features of sediments in conjunction with the method of experimental modeling of fireplaces makes it possible to establish the temperatures of previous heat exposures in the underlying substrate (Carrancho, Villalaín, 2011; Lagunilla et al., 2019), and fuel type from the magnetic properties of the ash (Peters, Church, Mitchell, 2001; Peters et al., 2002).

In the last ten years, methods of gas chromatography mass spectrometry (GC-MS) and X-ray fluorescence analysis (XRF) have been used as tools for studying pyrogenic objects at archaeological sites (Braadbaart et al., 2017). The GC-MS method of analysis establishes the chemical composition of organic substances and materials through the chromatographic separation of complex mixtures, with subsequent mass spectrometric detection. Identifying the ratio of organic compounds (alkanes, alcohols, sterols) serving as biomarkers of plant residues in pyrogenic samples makes it possible to suggest the type of fuel used for making the fire (Han, Calvin, 1969). The XRF method establishes the inorganic composition of deposits and archaeological materials for reconstructing the conditions of their emergence, the dynamics of the environment, and the correlation of horizons.

This article describes the study of complex burnt deposits at the stratified site of Surungur, carried out to establish their genesis (natural fires, controlled burning, fireplaces), as well as the types of fuel that were used by the humans during their habitation period at the site.

Materials

In 2017, the members of the Russian-Kyrgyz archaeological expedition in the Fergana Valley in Southern Kyrgyzstan discovered the multilayered Surungur site (Fig. 1). The study of the site gives some insights into the cultural dynamics and processes of human settlement in the Fergana Valley during the Holocene (Shnaider et al., 2021).

The Surungur site, located under a rock shelter made of limestone boulders, was studied in 2018–2019 (Olenchenko et al., 2019; Shnaider et al., 2021). Excavations were carried out over an area of 0.8 × 1.5 m, to a depth of 2.7 m.

The site has three main layers (the description is provided from top to bottom along the section) (Fig. 2). Layer 1 (1 m thick) is silty loams; broken debris from the roof of the shelter (limestone) is absent. Eight large

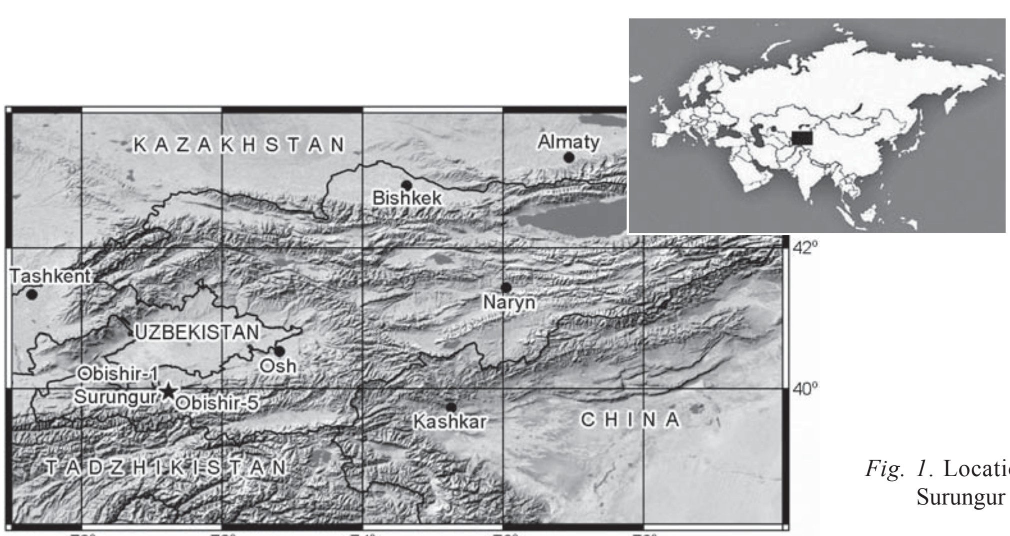

Fig. 1. Location of the Surungur site.

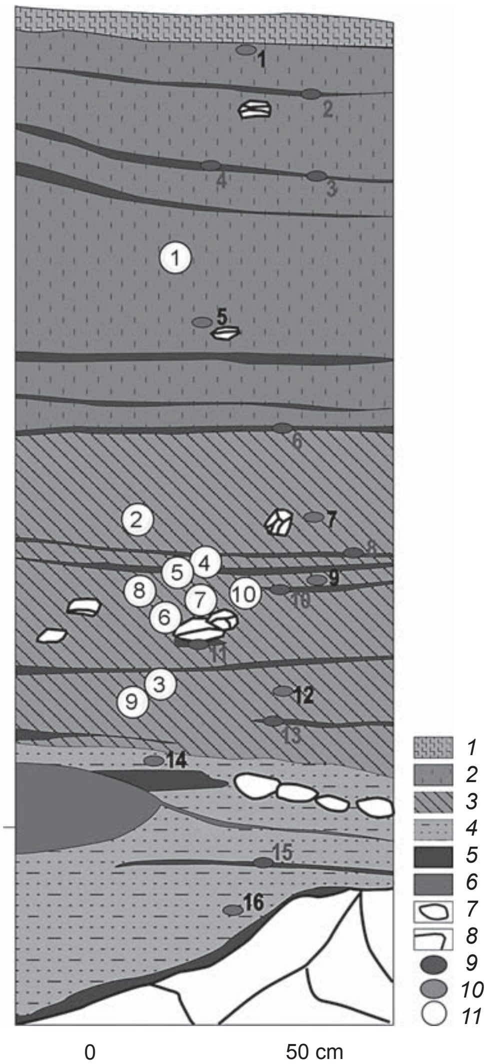

Fig. 2. Stratigraphic profile of the site.
1 – concrete; *2* – layer 1; *3* – layer 2; *4* – layer 3; *5* – ash-containing interlayer; *6* – interlayer of burnt sandy loam; *7* – debris; *8* – large stones on bedrock surface; *9* – place of sampling from ash horizons; *10* – place of sampling from inter-ash layers; *11* – place of sampling for absolute dating.

calcined spots were found, along with small fragments of burnt bones and pottery from the Chust culture of the Bronze Age. The boundary with the underlying layer was indistinct; it was identified from changes in density and the amount of debris. Layer 2 (from 0.6 to 0.8 m thick) was gray-brown silty loams; limestone debris was found in the middle part of the layer. Six calcined spots were discovered, along with numerous lithic artifacts and animal bones, including those showing traces of processing. The boundary with the underlying layer was also indistinct; it was identified by changes in the density and color of the deposits. Layer 3 (from 0.25 to 0.7 m thick) was brown silty loam; limestone debris occurred in small quantities. Three calcined spots were found, including a thick layer of burnt clay whose genesis is a subject of discussion and requires further field research; burnt pebbles lay nearby. The results of trace analysis have revealed that earlier the pebbles were a part of the lining on the hearth and could have been displaced by post-depositional processes. Scarce bone fragments were found in that layer. The rocky surface lay below.

First time, the platform under the stone ledge was inhabited in the Early Holocene. Indirect evidence on the presence of gramineous plants and long-term habitation of herbivores under the stone ledge was found in the sediments of the Middle Holocene. Osteological evidence from this period is dominated by the bones of the Ovicarpines. According to scholars, the industry of the site belongs to the Hissar Neolithic culture, which had not been found previously in the region (Shnaider et al., 2021).

During the works, 17 ash-bearing interlayers (burnt deposits) were identified: six in layer 1, eight in layer 2, and three in layer 3. Their thickness ranged from 1 to 7 cm; length ranged from 40 cm to 1 m. Individual large fragments of charcoal were found along the section. For laboratory studies, samples 2–4, 6, 8, 10, 11, 13, and 15 were taken from the central areas of nine distinct ash horizons in all three cultural layers (Fig. 2). These were taken from the central, thickest part of ash layers in the stratigraphic section of the site. In the layers without visible traces of thermal impact, areas with the highest values of magnetic susceptibility were identified using a KT-5 kappameter, and samples were taken from them. For establishing the degree of ash contamination in the cultural deposits of the site, samples 1, 5, 7, 9, 12, 14, and 16 were taken from the inter-ash layers (Fig. 2). Ten samples were taken for absolute dating: one from layer 1 and nine from layer 2.

Methods

Absolute dating

Radiocarbon analysis was conducted in the Accelerator Mass Spectrometry Center for Collective Use at Novosibirsk State University – Novosibirsk Scientific Center (AMS Golden Valley). Four bone fragments (GV-02123, GV-02797, GV-02798, and GV-02799), three soil samples (GV-02124, GV-02589, and GV-02590), and three samples consisting of a mixture of soil and charcoal (GV-02588, GV-02800, and GV-02801) have been analyzed. The bone fragments were very poorly preserved and probably burnt, with collagen content of no more than 1 %. Collagen could be extracted only from samples GV-02123 and GV-02798; humus was also extracted from GV-02123; only humus was extracted from GV-02797; and humus and charcoal were extracted from GV-02799. Humus was extracted from soil samples GV-02124, GV-02589, and GV-02590, and humus and charcoal were extracted from samples of the mixture GV-02588, GV-02800, and GV-02801. Each material—collagen, charcoal, and humus—was dated separately.

Samples were prepared in the Isotope Research Laboratory at the Institute of Archaeology and Ethnography SB RAS, using the standard methodology (Brock et al., 2010; Brock, Higham, Bronk, 2010). Carbonization of the obtained samples of each material was carried out using an absorption-catalytic unit (Lysikov et al., 2018); radiocarbon content was established using a unique research unit "Accelerator Mass Spectrometer of the Institute of Nuclear Physics of the Siberian Branch of the Russian Academy of Sciences" (Parkhomchuk, Rastigeev, 2011).

Rock-magnetic analysis

The following magnetic parameters have been established for the samples under study: magnetic susceptibility at room temperature at various frequencies (χFD), temperature dependence of magnetic susceptibility ($\kappa(t)$), anhysteretic remanent magnetization (ARM), saturation isothermal remanent magnetization (SIRM), hysteresis parameters, and parameter quantifying the contribution of magnetic minerals of different coercivity (S-ratio). That set of measurements was carried out in the Laboratory of the Main Geomagnetic Field and Petromagnetism at the Institute of Physics of the Earth RAS according to the standard methodology (Evans, Heller, 2003), under conditions similar to those adopted in the study of fires on loess substrate (Kulakova et al., 2021). Measurements were done in 3–4 duplicates, with subsequent averaging. The scatter of duplicate values was insignificant, and corresponded to normal distribution of the random variable.

Gas chromatography mass spectrometry method

Samples by volume 2–3 g were taken for analysis. Their extraction was carried out using a mixture of dichloromethane-methanol in a ratio of 9:1 (the volume of 5 ml), with the addition of an internal standard (1 mg of biphenyl) in sealed vials at the temperature of 80 °C for three hours. After cooling, the solution was filtered and solvent was purged with dry nitrogen to the volume of 100 μl.

The prepared samples were analyzed using a GC-MS-system Agilent 7000B (made in the USA) based on three quadrupoles. For separating the substances contained in the samples, an HP-5ms capillary column (30 m × 0.25 mm × 0.25 μm) was used. The analysis was performed under the following conditions: the oven temperature program from 130 to 310 °C at a rate of 11 °C/min; inlet temperature 310 °C, split 5:1; carrier gas (helium) flow rate 1.2 ml/min. Mass spectrometer was used in scanning mode in the mass range of 40–500 m/z; ionization energy 70 eV; the ionization source temperature 250 °C.

Quantitative analysis was carried out according to the internal standard (biphenyl). Sensitivity coefficients of biphenyl to n-alkanes and alcohols were established by analyzing a mixture of biphenyl, docosane, and dodecanol standards.

X-ray fluorescence analysis

All samples from the Surungur site were mixed and averaged by the quartering method. Each selected sample (0.250 g) was ground for several minutes in an agate mortar, after which a sample of diluent (polyethylene), chemically pure and transparent in the X-ray range and weighing 0.750 g, was added to it. The mixture, weighing 1 g, was thoroughly mixed again until it became homogeneous, after which a tablet was formed of it using an Atlas T25 (Speciac) automatic press under a load of 21 tons.

The composition of the tablet was established using an ARL Perform'X X-ray fluorescence spectrometer (Thermo Scientific) with the Rh-anode tube. The content of elements was calculated using the UniQuant software.

Since the samples were a mixture of host rock and ash, the content of the identified components in the surrounding soil had to be taken into account for reliably obtaining the chemical composition of the combustion products. Sample 14, which was a pure material of the host rock, was used as reference. The ratio of the main components (Al and Si) calculated for it was applied to further analysis of the chemical composition of the ash.

Results

Absolute dating

The results of radiocarbon dating of three types of samples—bone collagen, charcoal, and humus—indicate that people settled at the site during the Holocene period (Fig. 3). There is a linear correlation between the age and depth where the samples occurred, which suggests a fairly uniform sedimentation in the region under study over the last 7000–8000 years. A date from bone and humus was obtained for layer 1; it indicates that the middle part of the layer emerged ca 3000–3500 BP. For layer 2, a series of dates was obtained that fit the chronological range of 7900–5900 BP (Fig. 3). Pieces of charcoal (samples GV-02800 and GV-02588), located in the section in close proximity

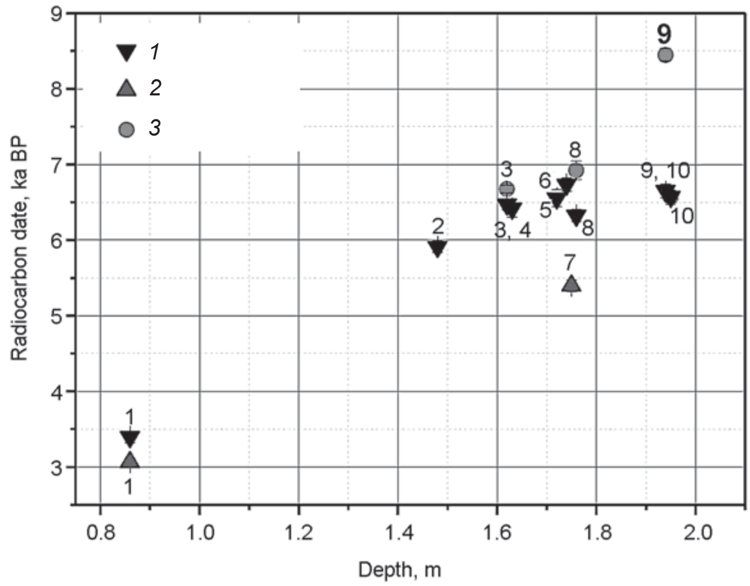

Fig. 3. Graph of the dependence of dates on the depth of samples.
1 – humus; *2* – collagen; *3* – charcoal.

to each other, reveal a large difference in age of ca 1800 years, which can be explained by the fires that happened at that time and involved ancient sites, and mixing of soil layers during the period of intense habitation at the site.

Rock-magnetic analysis

As opposed to the pyrogenic objects of natural origin, the objects of anthropogenic origin are distinguished by higher temperatures resulting in increased concentrations of magnetite (Kulakova et al., 2021; Jrad et al., 2014). The rock-magnetic method was used for studying sixteen samples from the section of the Surungur site (Table 1). Three of the samples taken from the inter-ashy layers—1, 14, and 16—have revealed similar magnetic properties corresponding to the lowest values of rock-magnetic parameters. Temperature curves of magnetic susceptibility in these samples show the greatest differences in the initial and final values of magnetic susceptibility after the cycle of heating/cooling reaching 700 °C (Fig. 4). The cooling curve was significantly higher than the heating curve, indicating predominant formation of magnetite during the temperature impact (according to the Curie point). The values of rock-magnetic parameters and behavior of the thermal curves indicate that samples 1, 14, and 16 did not experience significant heating effect in the past and do not contain ash; therefore, they can be considered to be host rocks. As opposed to other samples, these samples were characterized by the lowest values of the S-ratio$_{100mTl}$ (0.79–0.86); this suggests a relatively higher content of hematite/goethite relative to magnetite/maghemite, and may reflect the initial ratio of the magnetic minerals in host rocks. In appearance, sample 14 stands out among the others by its reddish tint, which may be associated with higher concentration of goethite.

The remaining thirteen samples corresponded to the values of magnetic susceptibility from 1.37 to 5.2×10^{-6} m^3/kg; values of frequency dependence of magnetic susceptibility ranged from 12.0 to 49.3× ×10^{-8} m^3/kg; the SIRM values ranged from 12.3 to 35.7 mAm^2/kg, and the ARM values from 0.11 to 0.39 mAm^2/kg. This indicates the increased concentration of magnetic minerals. According to the hysteresis data, magnetite was the main magnetic mineral in these samples. The presence of hematite and goethite was more typical of the composition of samples 5, 9, and 12 from inter-ash layers. The increased concentration of magnetite/maghemite is associated with the presence of either a thermally altered substrate or ash.

Relative ash content was estimated from the difference between the initial values of magnetic susceptibility of the samples under consideration and host rocks on the temperature curves (Fig. 4). If the initial values of magnetic susceptibility on the thermal curves corresponded to those of the host rocks, the sample most likely did not contain ash. The relative ash content in the rest of the samples was conventionally identified as low (the increase in magnetic susceptibility up to 2 times), significant (2–3 times higher), or high (more than 4 times higher).

Gas chromatography mass spectrometry analysis

The analysis has revealed the traces of C_{20}-C_{33} n-alkanes, and C_{20}-ol, C_{22}-ol, C_{24}-ol, and C_{26}-ol even

Table 1. **Petromagnetic values of the samples from the Surungur site**

Sample	χ, × 10^{-6} m^3/kg	$χ_{FD}$, × 10^{-8} m^3/kg	SIRM, mA m^2/kg	ARM, mA m^2/kg	S-ratio	
					100 mTl	300 mTl
SR-1	0.97	8.1	8.3	0.09	0.81	0.96
SR-2	2.95	27.9	22.2	0.24	0.93	0.99
SR-3	1.80	16.4	15.1	0.16	0.92	0.99
SR-4	1.37	12.0	12.3	0.11	0.89	0.98
SR-5	2.53	26.2	16.2	0.18	0.84	0.99
SR-6	3.06	26.2	26.6	0.29	0.90	0.99
SR-7	5.20	49.3	35.7	0.39	0.94	0.98
SR-8	2.71	25.4	21.7	0.23	0.90	0.99
SR-9	1.40	12.2	12.4	0.12	0.89	0.99
SR-10	2.98	27.0	23.5	0.27	0.92	0.99
SR-11	3.87	36.9	28.4	0.31	0.93	1.00
SR-12	2.73	24.7	21.6	0.22	0.91	1.00
SR-13	2.46	24.2	17.8	0.22	0.89	0.98
SR-14	1.06	9.0	8.8	0.09	0.79	0.92
SR-15	3.31	31.6	24.1	0.28	0.90	0.99
SR-16	0.99	9.0	8.9	0.10	0.86	0.98

alcohols in the samples. Bacteria, algae, and plants are the source of high molecular weight of alkanes and alcohols (Han, Calvin, 1969). The typical features of higher plants include a significant predominance of odd over even n-alkanes, as well as the presence of even alcohols and the complete absence of odd alcohols.

For quantitative estimation of predominance degree of odd n-alkanes over even n-alkanes and thus for classification of sediments according to the presence of higher plant residues in them, it was proposed to use the OEP index, which takes into account C_{26}–C_{33}, that is, the main hydrocarbons that are a part of plant epicuticular waxes (Zech et al., 2009). The ratio between n-alkanes C_{27}, C_{29}, and C_{31} for different life-forms of plants does not coincide. It has been suggested that n-alkanes C_{27} and C_{29} dominate in most modern trees and shrubs, while n-alkanes C_{31} and C_{33} dominate in grasses (Ibid.). The $C_{31}/(C_{29} + C_{31})$ ratio is used for numerical characterization of such dependences (Bush, McInerney, 2013). As for alcohols, they can be considered an additional trait to n-alkanes, corresponding to higher plants.

The analysis has revealed significant differences between the samples in the content of alcohols and n-alkanes (Table 2). Their highest content was in samples 1, 2, 4, 7–9, and 11. In contrast, sample 15 had a very low content of alkanes (0.01 mg/kg), which was an order of magnitude lower than the values in other samples. Although the total content does not provide the necessary information, it is important to mention that samples 1 and 2, which contained the largest amount of alkanes, were taken relatively close to the surface, where grasses grow. Therefore, upon further consideration, we believe that any conclusions about plant-remains from these two samples should be drawn with extra caution.

The OEP-index for samples 5, 7, 9, and 16 was less than 1, which indicates the absence of higher plant-residues in these samples and the microorganism origin of the observed biomarkers. These samples do not belong to ash interlayers; therefore, they are not to be considered while assessing the type of fuel used. As far as the index $C_{31}/(C_{29} + C_{31})$ is concerned, samples 1, 2, and 8 showed values over 0.6; consequently, the observed biomarkers were most likely of herbal origin. Samples 1 and 2 were probably contaminated by grasses from the surface, and only sample 8 can be attributed to the remains of grass.

The $C_{31}/(C_{29} + C_{31})$ index for the remaining samples (3, 4, 6, and 10–15) was close to 0.5. This indicated the mixed origin and use of mixed fuels with some

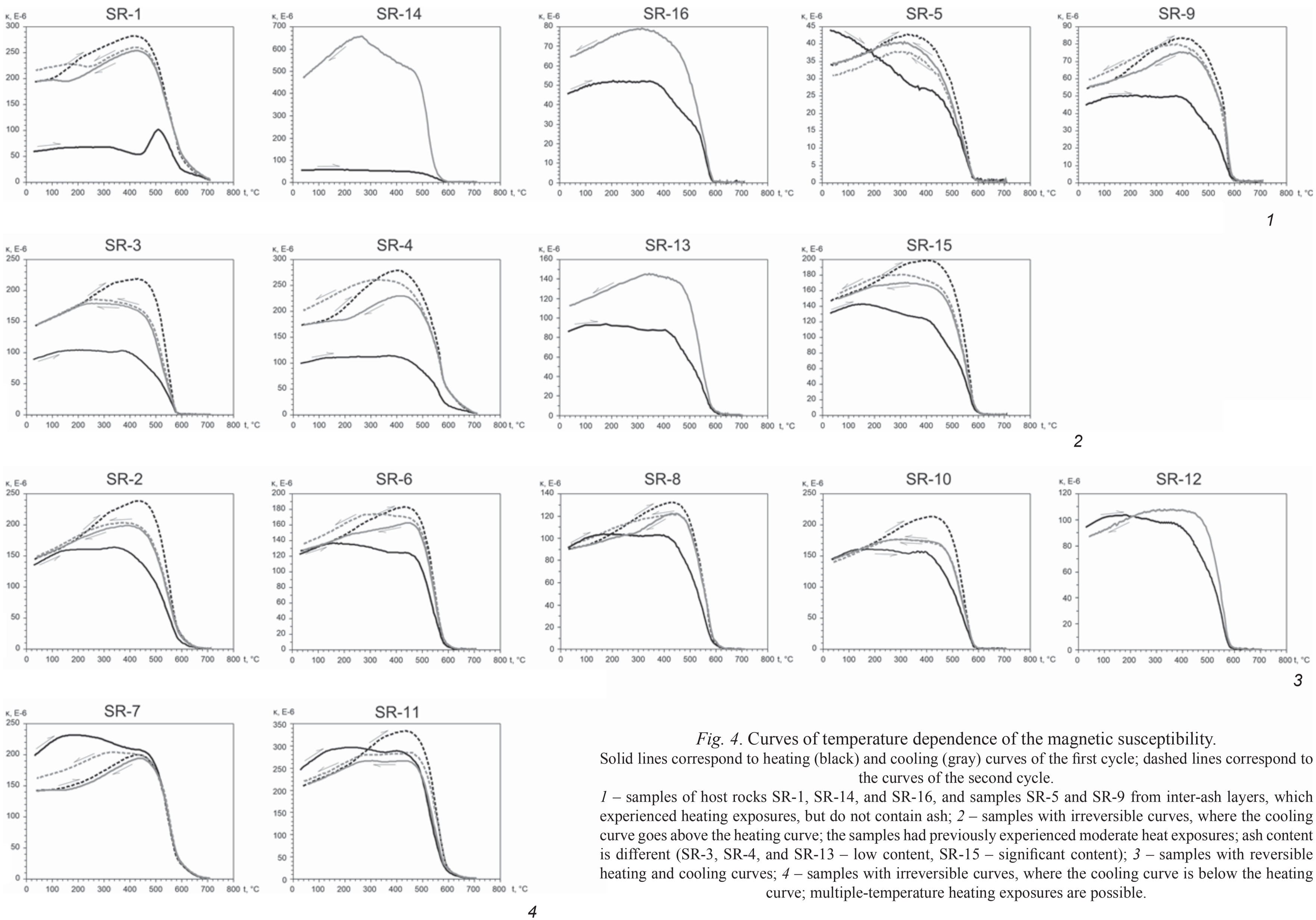

Fig. 4. Curves of temperature dependence of the magnetic susceptibility.
Solid lines correspond to heating (black) and cooling (gray) curves of the first cycle; dashed lines correspond to the curves of the second cycle.
1 – samples of host rocks SR-1, SR-14, and SR-16, and samples SR-5 and SR-9 from inter-ash layers, which experienced heating exposures, but do not contain ash; *2* – samples with irreversible curves, where the cooling curve goes above the heating curve; the samples had previously experienced moderate heat exposures; ash content is different (SR-3, SR-4, and SR-13 – low content, SR-15 – significant content); *3* – samples with reversible heating and cooling curves; *4* – samples with irreversible curves, where the cooling curve is below the heating curve; multiple-temperature heating exposures are possible.

Table 2. **The total content of n-alkanes and high molecular weight alcohols, as well as generalized indices in the samples from the Surungur site**

Sample	Alkane content, mg/kg	Alcohols content, mg/kg	$C_{31}/(C_{29} + C_{31})$	CPI	OEP
SR-1	6.57	0.28	0.61	7.135	8.822
SR-2	1.68	0.14	0.834	5.413	6.966
SR-3	0.04	0	0.479	2.377	3.637
SR-4	0.89	0.14	0.501	1.081	1.45
SR-5	0.1	0.09	0.408	0.916	0.913
SR-6	0.03	0.01	0.476	1.678	2.442
SR-7	1.81	0.33	0.281	0.809	0.659
SR-8	0.88	0.09	0.613	1.26	1.565
SR-9	0.6	0.77	0.225	0.832	0.526
SR-10	0.16	0.02	0.531	4.166	5.599
SR-11	0.67	0.09	0.425	1.613	1.893
SR-12	0.07	0.01	0.509	3.776	4.229
SR-13	0.06	0	0.525	1.683	2.327
SR-14	0.08	0.03	0.557	1.483	1.71
SR-15	0.01	0	0.483	1.404	1.696
SR-16	0.23	0.29	0.447	0.999	0.969

prevalence of wood content for samples 3, 6, 11, and 15 ($C_{31}/(C_{29} + C_{31})$ – index less than 0.5).

X-ray fluorescence analysis

The compounds of silicon, calcium, phosphorus, and potassium can be considered to be the most pronounced markers of the type of fuel among inorganic components available for analysis (Table 3). As a rule, large content of silicon, as well as increased content of potassium and phosphorus, in ash suggests the predominant use of grass or dung as fuel. High content of calcium is typical for combustion products of wood species. Silicon was chosen as a marker, since the actual calcium content of samples might have been strongly influenced by the surrounding limestone rocks. Since the analysis of potassium and phosphorus did not reveal any regularities, which can be explained by uncertainty of the contribution of the host rock, these elements were not used for assessing the fuel type in this study.

Discussion

Rock-magnetic analysis

For interpreting the samples, with the exception of host samples 1, 14, and 16, a graph of the dependence of magnetic susceptibility (χ) on anhysteretic remanent magnetization ARM was made (Fig. 5). Since both parameters were concentration-dependent, we can speak of an increased concentration of magnetic minerals (represented mainly by fine-grained magnetite) from sample 4 to sample 7 (Fig. 5).

Samples 2–5 (layer 1) show lower concentrations of magnetic grains as compared to samples from layer 2 (6–13) and the ash interlayer from layer 3 (15) (Fig. 5). An analysis of the thermomagnetic curves (see Fig. 4) allows the conclusion to be drawn that sample 5 (from the inter-ash space) doesn't contain ash, but experienced high-temperature heat exposures at an earlier time. The magnetic susceptibility of samples 3 and 4 continued to grow during the cycle of heating up to 700 °C and subsequent cooling. Accordingly, it can be concluded that the heat exposure of the substrate (possibly up to 400 °C) was moderate, and that a small amount of ash is present in the samples. Sample 2 shows reversibility of the heating and cooling curves, which suggests previous high-temperature heat exposures (600–650 °C); this sample probably contains a significant amount of ash. The unequal heat exposure of samples 2–4 can be associated with combustion of different types of fuel on them (Aldeias, 2017)

Table 3. **The content of the main inorganic components in the samples from the Surungur site, established by the XRF method (without taking into account the composition of the reference sample), wt%**

Sample	Na	Mg	Al	Si	P	S	Cl	K	Ca	Ti	Mn	Fe	Zn
SR-1	0.47	3.2	5.41	15.32	0.63	0.64	0.08	2.49	26.77	0.42	0.13	4.13	0.25
SR-2	0.37	4.87	4.62	18.86	1.17	0.21	–	2.65	21.3	0.32	0.12	3.37	0.17
SR-3	0.15	1.54	1.5	4.49	0.47	0.07	–	0.74	30.42	0.57	–	1.06	0.99
SR-4	0.5	3.45	4.79	14.44	1.89	0.19	0.08	2.31	28.62	0.34	0.11	3.28	0.13
SR-5	0.25	6.83	3.5	17.46	1.71	0.18	0.08	3.22	20.31	0.3	0.13	3.15	1.71
SR-6	0.23	5.26	2.8	10.09	0.83	0.1	0.07	2.32	17.99	0.2	0.1	2.02	0.89
SR-7	0.47	5.95	3.62	13.88	1.63	0.15	0.11	3.24	28.23	0.28	0.14	2.94	0.09
SR-8	0.4	5.52	3.26	12.47	1.11	0.11	–	2.8	31.95	0.33	0.27	3.3	0.36
SR-9	–	6.09	3.24	12.65	1.04	0.11	0.07	3.2	26.25	0.37	0.19	4.42	4.52
SR-10	0.07	5.72	3.59	12.96	1.05	0.09	–	2.79	28.91	0.42	0.16	3.76	2.15
SR-11	0.43	5.83	3.66	13.79	1.67	0.09	–	2.71	29.01	0.29	0.13	2.91	0.13
SR-12	0.26	4.62	2.59	9.24	0.9	0.06	–	2.12	19.76	0.21	0.1	2.47	0.53
SR-13	–	5.76	3.93	13.71	0.85	0.12	–	2.74	26.64	0.37	0.16	3.86	3.27
SR-14	–	3.33	6.19	14.79	0.22	–	–	3.18	11.87	0.46	0.1	4.61	2.56
SR-15	–	3	3.17	10.47	0.84	0.1	–	2.27	19.48	0.28	0.12	2.73	2.25
SR-16	–	3.09	3.16	11.25	0.58	0.1	–	1.85	19.17	0.27	0.1	2.87	2.02

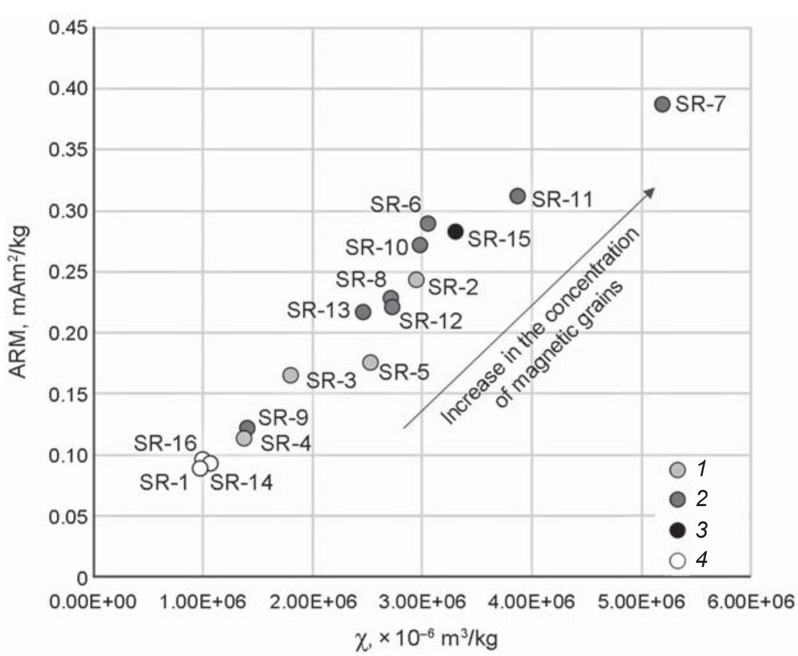

Fig. 5. Graph of dependence of magnetic susceptibility χ on anhysteretic remanent magnetization ARM.
1 – layer 1; *2* – layer 2; *3* – layer 3; *4* – host rocks without traces of previous heating and ash.

or with the location of the substrate at the periphery or in the center of the combustion area (Carrancho, Villalaín, 2011).

Samples 6–13 from cultural layer 2 manifest the highest concentrations of magnetic minerals (see Fig. 5). Sample 9 from the inter-ash space doesn't contain ash, but experienced moderate heat exposures (up to 500–600 °C) (see Fig. 4). Sample 13 contains a small amount of ash, but its magnetic susceptibility temperature curves are irreversible; most likely, it belonged to the peripheral area of the fireplace (probable instances of heat exposure up to 400–450 °C). Samples 6, 8, 10, and 12 exhibit a similarity in the behavior of the temperature curves and show their reversibility, which indicates previous heat exposures to more than 650–700 °C. The ash content in these samples is different: it is significant in samples 6 and 10, and low in samples 8 and 12. Samples 7 and 11, which correspond to the highest values of magnetic susceptibility and remanent magnetization, show decreased value of magnetic susceptibility when studying its temperature dependence. This is associated with high-temperature transformations (Maki, Homburg, Brosowske, 2006) and testifies to repeated fires made by ancient people in the same place. According to rock-magnetic data, these samples contain the largest amount of ash. Notably, the GC-MS-analysis has not revealed the remains of higher plants in sample 7, which can be explained by their complete burnout.

Sample 15 from the ash interlayer of cultural layer 3 shows high values of rock-magnetic parameters (see Fig. 5), but a slight excess of the cooling curve over heating curve on the temperature curves of magnetic susceptibility (see Fig. 4). Most likely, the sample is a thermally altered substrate from the area close to the central part of the fireplace (possible previous heat exposures up to 600 °C), with significant ash content.

Chemical analyses

According to the results of the GC-MS-analysis, most of the samples contain even alcohols and odd n-alkanes—markers of higher plants, with the exception of samples 5, 7, 9, and 16, which don't contain plant biomarkers.

Samples 1, 2, and 8 include mainly grass-residues; only in sample 8 can they be associated with human activity and type of fuel used.

Samples 3, 4, 6, and 10–15 show a mixed composition (grasses and wood). People probably used grasses and wood equally (recorded by the predominance of wood in samples 3, 6, 11, and 15).

The XRF analysis has shown that samples contain combustion products: mainly of grasses in 2, 7, 8, 10, and 13, and mainly of wood in 3, 6, 12, and 15 (Table 4).

Table 4. **Calculated silicon content in the ash of the samples from the Surungur site and the supposed type of fuel, according to the data obtained from the GC-MS and XRF analyses**

Sample	Si content in ash, wt%	Supposed type of fuel	
		According to the GC-MS data	According to the XRF data
SR-2	7.82	Predominantly straw/dung	Predominantly straw/dung
SR-3	0.91	Predominantly wood	Predominantly wood
SR-4	2.99	Mixed type	"
SR-5	9.10	No plant remains	Predominantly straw/dung
SR-6	3.4	Predominantly wood	Mixed type
SR-7	5.23	No plant remains	"
SR-8	4.68	Predominantly straw/dung	"
SR-9	4.91	No plant remains	"
SR-10	4.38	Predominantly straw/dung	"
SR-11	5.04	The same. Possible contamination with debris	"
SR-12	3.05	Predominantly wood	"
SR-13	4.32	Predominantly straw/dung	"
SR-15	2.9	Predominantly wood	"

Note: "Mixed" type of fuel means equal content of combustion products of wood/shrubs and grasses/animal dung.

The data obtained from the GC-MS and XRF analyses, as revealed by their comparison, are in good agreement with each other, with the exception of sample 11. Sample 11 is most likely contaminated, since it was taken in a place adjacent to the zone of debris (see Fig. 2).

Comparison of the results of rock-magnetic and chemical analyses has shown that the samples with high ash content yielded traces of exposure to high temperatures (Table 5). Consequently, there were long-term and non-isolated acts of burning plant material. In this case, the chemical composition indicates mainly the use of a mixed type of fuel, but the temperature of the heat does not depend on the type of fuel. Ash layers in cultural layers 1 and 2 contain mainly the remains of burning grasses and/or animal dung, as well as wood. In layer 3, the fuel for the pyrogenic object was wood, and, to a lesser extent, grasses.

Conclusions

During the field study of site deposits, ash horizons alternating with loam deposits have been found. Radiocarbon analysis of archaeological evidence (bones and charcoal) has revealed that humans settled at this site in the Early Holocene.

According to the rock-magnetic parameters (magnetic susceptibility, frequency magnetic susceptibility, ARM, and SIRM) and the data on the paleotemperature heat exposures of samples 1–13, cultural layers 1 and 2 were subjected to high temperatures for a long time; the highest and multiple heat exposures (above 650–700 °C) were found in the samples from the deposits of cultural horizon 2. Using the GC-MS and XRF methods, it has been established that the source of long-term high-temperature exposures during this period could have been woody species of deciduous trees, grasses, shrubs, and dung. Sample 1 from the roof of layer 1 was not exposed to direct fire, since it was not contaminated by combustion products. The soil corresponding to samples 5 (layer 1) and 9 (layer 2) had been subjected to significant temperature effects in the past, but was not contaminated with ash.

Cultural layer 3 differs in its composition (features) from the overlying layers. Only three ash-containing interlayers have been found in it; the enclosing soil was not contaminated with ash (samples 14 and 16). This can be explained by short-term habitation of humans under the stone ledge, since ash from the fireplaces should have been mixed with the soil under prolonged anthropogenic impact. One pyrogenic object (sample 15) from the middle of the layer was examined. According

Table 5. **Results of studying the samples from the Surungur site**

Sample	Prolonged heat exposures of soil, t, °C	Ash content	Type of fuel according to the GC-MS and XRF data
SR-1	–	–	–
SR-2	600–650	Significant	Mixed
SR-3	Up to 400	Not too high	
SR-4	Up to 400	"	More wood
SR-5	> 600	–	–
SR-6	> 700	Significant	More wood
SR-7	Multiple > 700	Very high	More straw/dung
SR-8	> 700	Not too high	
SR-9	500–600	–	–
SR-10	> 700	Significant	More straw/dung
SR-11	Multiple > 700	Very high	
SR-12	> 700	Not too high	More wood
SR-13	400–450	"	More straw/dung
SR-14	–	–	–
SR-15	Up to 600	Significant	More wood
SR-16	–	–	–

to rock-magnetic data, the deposits in this area were exposed to high temperatures (up to 600 °C): they contain a significant amount of ash, which points to the anthropogenic origin of the ash layer. According to the results of the GC-MS and XRD analyses, people used mainly wood and, to a lesser extent, grasses.

On the basis of the data of multidisciplinary study, it is possible to have an idea of the stages of human settlement at the Surungur site. During the formation of layer 3, people lived under the rock shelter several times, using wood and grasses as fuel. The time when layer 2 was formed corresponds to the stage of the most active and constant stay of people on the platform under the stone ledge. This is evidenced by the maximum values of heat effect on the deposits and extremely high ash-content in the cultural layer. During this period, the inhabitants of the site used mainly grasses and/or animal dung and wood as fuel. The accumulations of layer 1 probably reveal short-term events of human habitation in the platform under the stone ledge; heat effects on the deposits were not as significant. Grass/animal dung and wood were used as fuel. This study makes it possible to conclude that woody vegetation was available in the area of the site during the period of human habitation there.

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