ISSN 1064-2293, Eurasian Soil Science, 2025, Vol. 58:36. © Pleiades Publishing, Ltd., 2025.

MINERALOGY AND MICROMORPHOLOGY OF SOILS

# Micromorphological Features and Formation of the Cultural Layer in the Early Medieval Town of Dzhankent (Eastern Aral Sea Region, Kazakhstan)

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Received June 21, 2024; revised November 5, 2024; accepted November 5, 2024

**Abstract**—This paper presents the results of a study on the stratigraphy, morphology, micromorphology, and chemical and physicochemical characteristics of the cultural layer in the residential area of the Early Medieval town of Dzhankent (Eastern Aral Sea region, Kazakhstan). The town is located in an arid paleodeltaic landscape with a dynamic water supply. Sets of diagnostically significant micromorphological features are described, including: (1) features resulting from anthropogenic input and redistribution of matter (plant detritus, phytoliths, bone fragments, etc.); (2) pedofeatures resulting from the transformation of human-introduced material (pyrogenic forms of carbonates, organic and phosphate-organic pedofeatures); and (3) pedofeatures consisting of carbonates, gypsum, and iron oxides, mainly associated with natural processes. Stratigraphic units of the cultural layer, along with the processes and conditions of their formation, were defined, and the archaeological context was interpreted for each lithostratigraphic unit based on their morphological and analytical characteristics. The upper stratigraphic unit consists of the destruction products of clayey adobes, with ash interlayers, some of which are stratigraphically traceable and may correspond to fire events. The middle unit comprises residential and domestic sediments, including a series of living surfaces, and contains an ash layer possibly resulting from a fire event. The lower unit is composed of alluvial-deltaic deposits slightly impacted by residential activities and periodically affected by fluctuating groundwater. The distribution of organic carbon and phosphorus in the cultural layer correlates well with the observed organic substrates at the macrostratigraphic level, being significantly higher in loose layers rich in plant detritus, charcoals, ash, and bones. However, the analytical characteristics of the cultural layer (pH, electrical conductivity, contents of organic carbon, phosphorus, carbonates, and gypsum), determined from homogenized samples of the stratigraphic units, do not always align with the composition observed at the micromorphological level (abundance of micro-artifacts, anthropogenic and natural microfeatures). These discrepancies are primarily due to the extremely high lateral and microstratigraphic heterogeneity of the cultural layer.

**Keywords:** micromorphology of a cultural layer, anthropogenic microfeatures, natural microfeatures, Urbic Technosols

**DOI:** 10.1134/S1064229324604141

## INTRODUCTION

Human activity at different times can result not only in the creation of objects and artifacts of material culture, but also in the alteration of the environment, reflected in the transformation of paleolandscapes, in the pollution of natural environment by household and industrial waste, in the transformation of the structure and chemical and physicochemical properties of soils and sediments, and in the emergence of specific anthropogenic soil and sediment strata associated with human settlements and economic activity. Cultural layers are the product of transformation of the original, natural parent organic or mineral substrate by a complex of natural and anthropogenic processes [2, 36]. The processes associated with the accumulation of natural and anthropogenic substrates and their subsequent transformation by soil processes can be of mechanical, physicochemical, chemical, and biological provenance. Some of them operate only at the stage of accumulation of the cultural layer, while others continue to function and come to the fore during periods of temporary waning of life in settlements, or at the postanthropogenic stage, if the settlement has completely ceased to exist. The concurrent or phased contribution of natural and anthropogenic processes to the development of cultural layers stipulates the need for an integrated approach to their study and the high significance of such studies both for the Earth sciences (geography and paleogeography, Quaternary geology, soil science) and for the humanities, particularly archaeology. The study of natural features provides insight into the landscape and climatic context of the formation and transformation of cultural layers. The variety of anthropogenic features offers a wealth of additional information for interpreting the genesis of the layers and the nature and intensity of anthropogenic impacts in the past [2, 35, 38, 40, 46, 50].

The understanding of the cultural layer as a product of interaction between humans and the environment, the value of this object for paleoecological studies, and the possibility of using soil approaches and methods in such studies were actively pursued in the 1980s and 1990s [19, 20, 46]. In Russian studies of cultural layers of settlements, great attention traditionally has been paid to their morphology, physicochemical and chemical properties [4, 6, 21, 22, 37, etc.], as well as to the comparative analysis of microfeatures and processes in cultural layers and natural soils [25, 31, 33]. Great importance is attached to the results of comparative analysis of cultural layers of archaeological sites that functioned under different natural conditions [23, 51], and to the reconstruction of regional paleogeographic conditions and presettlement stages of soil formation, including the studies of different functional zones of settlements [3, 6, 11, 15, 43]. Foreign studies of cultural layers of settlements mostly focus on the reconstruction of regional paleogeographic conditions of ancient settlements [41], on the study of the anthropogenic impact on paleoecosystems [54, 55, 61, 62,], and on the reconstruction of human settlement activity in the past [47, 49, 58, 60, 68]. Some papers explore the general nature of the cultural layer as a natural–anthropogenic product, and the processes of formation and transformation, assessing preservation of anthropogenic features in the postanthropogenic period [2, 5, 34, 35, 48].

There are many questions, still to be looked into, regarding patterns in the formation of cultural layers of ancient settlements, the set of natural and anthropogenic processes that shape them, and the relationships between these processes, on the one hand, and the conditions of the natural environment and the nature of anthropogenic impacts, on the other. The micromorphology applied to archaeological sites over the past two decades has become an advanced tool in microstratigraphic studies of cultural layers in aid of archaeological interpretations [59, 64]. There is a need to find a universal method that would help to understand the genesis, processes of formation and transformation of cultural layers. However, micromorphological studies have not yet received sufficient recognition in addressing issues of genesis, and the processes of formation and transformation of cultural layers. This paper presents the results of the study of microstructure, chemical and physicochemical properties of a complete stratigraphic column of the thick cultural layer of the Dzhankent ancient settlement. Located in the Eastern Aral Sea region in the arid landscape of the Syr Dar'ya paleodelta, the site is a large Early Medieval urban settlement, built in the technique of earthen architecture, with a developed infrastructure and varied agrarian, handicraft, and trade economic activities.

The purpose of the work is to diagnose anthropogenic and natural soil processes and conditions for the formation of the cultural layer of the Dzhankent settlement, based on stratigraphy, macro- and micromorphological features and analytical characteristics of the site.

## OBJECTS AND METHODS

The study area is located in the Turan Lowland, on the flat delta-alluvial plain of the Syr Dar'ya River (northwestern part of Kyzylorda oblast, Republic of Kazakhstan) (Fig. 1a). The climate is arid and strongly continental with considerable daily and seasonal temperature fluctuations. The January temperature is −11.3°C, that of July, +26.0°C, and the mean annual temperature is +8.0°C. Annual precipitation is below 120 mm and evaporation is 1500 mm [29]. The parent material for present-day natural soils in the area, as well as the lithological basis for the formation of the cultural layer are unconsolidated ancient alluvial and deltaic sediments of the Syr Dar'ya River with the layering represented by alternation of silty clays, loams, sandy loams, and fine-grained silty sands [12]. The present-day vegetation cover consists predominantly of tamarisk and wormwood and sagebrush plant associations. The soil cover in the study area is represented mainly by gray-brown, meadow-bog, takyr-like soils, takyrs, and desert sandy and saline soils [13]. The site area has a complex natural hydrological network consisting of functioning and dry delta channels, streams, and lakes, overlaid by a set of modern irrigation hydraulic engineering constructions, most of which are not functioning. According to the machine drilling performed in August 2018–2019, the groundwater table in the area of the site is about 8 to 9 m below the daylight surface.

The research was carried out in the area of the Early Medieval settlement of Dzhankent (Fig. 1b). The site is one of the "marsh towns" described and preliminarily investigated by the Khorezm expedition in 1946 [9]. In its heyday, the settlement was a large fortified urban settlement, a trade and craft center on the intersection of the Northern Silk Road with the route from the Volga River to Khorezm. The site is surrounded by earthen (pakhsa[1]) town walls. The settlement within

[1] Pakhsa is rammed clay used as a building material in Central Asia [17].

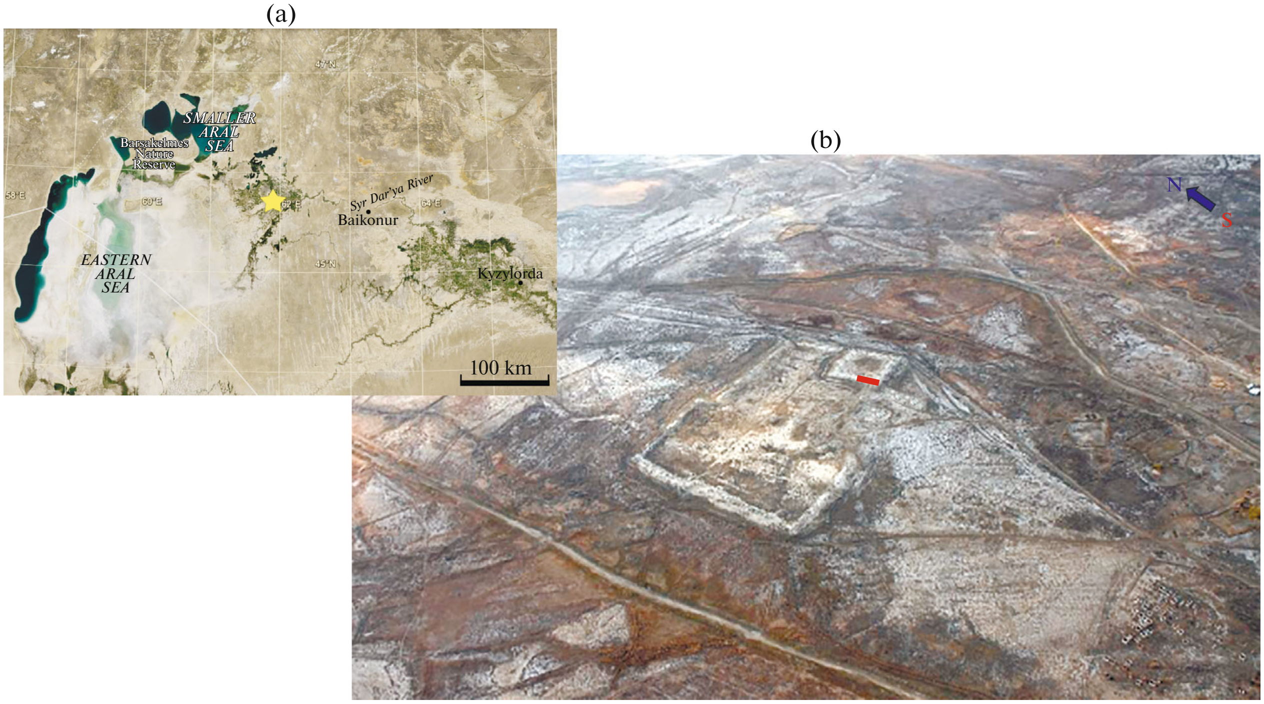


**Fig. 1.** Location of the Dzhankent settlement: (a) a fragment of the map of the Republic of Kazakhstan (location of the site is marked with a symbol); (b) an aerial photograph of the site (the symbol marks the location of the profile under study) [8].

the walls is about 16 hectares in area with a clear layout. The cultural layer is represented by stratified beds up to 9 m thick. The surviving architectural fragments are mainly mudbrick masonry and earthen structures. The archaeological research of the cultural layer inside the residential quarters of the settlement discovered evidence of nonferrous metals processing, pottery production, and cattle husbandry. It is assumed that the decline of the settlement was due to the general political situation in the region and, possibly, the migration of the Syr Dar'ya river channel [9].

In 2012, a cross-section was made through the Northern defensive wall of the settlement at the junction of the Citadel wall and the general defensive wall of the town. As the archaeological excavations showed, this wall was heterogeneous in its composition and had undergone several repairs and rebuildings. The later wall made of mudbricks and pakhsa blocks was built on top of the earlier habitation layers rich in animal bones, pottery fragments, and other archaeological material. Therefore, the layers inside the "body" of the wall were not formed at one time during construction, but rather had accumulated over a long period of time as a result of life activity of the town's inhabitants [9]. It was these residential layers, which were adjacent to the inner part of the wall in the early stages of settlement and were subsequently sealed into the body of the town wall later during the rebuilding, that served as the object of the study (Fig. 2).

The study applied a hierarchical morphogenetic approach along with a set of physicochemical analyses. Micromorphological examinations of samples were performed in thin sections using a Nikon E200 Pol polarizing microscope at magnifications of 40×, 100×, and 400×. Micromorphological descriptions, diagnostics and interpretation of features were performed using both domestic and international approaches [18, 53, 63, 66]. We performed an experience-based semiquantitative assessment of the occurrence of microfeatures in layers in the micromorphological analysis. The occurrence of microfeatures was semiquantitatively assessed in each thin section corresponding to a certain stratigraphic layer on a 4-point scale: 4, many; 3, common; 2, few; 1, single; and 0, absent. The results of the semiquantitative assessment are presented in Table S3.

We used standard methods to study the physicochemical properties [10, 16, 28]: the pH of the water extract was determined by the potentiometric method; total phosphorus by the Ginzburg method (wet digestion with potassium bichromate in the presence of sulfuric acid), with photometric termination according to Kirsanov as modified by laboratory of the Institute of Geography, Russian Academy of Sciences; carbonates by alkalimetric titration; organic carbon by Tyurin's method modified by Nikitin with photometric termination (wet digestion with potassium bichromate in the presence of sulfuric acid); and gypsum by gravimetric method; electrical conductivity of the water extract was measured with conductometer KP-00.

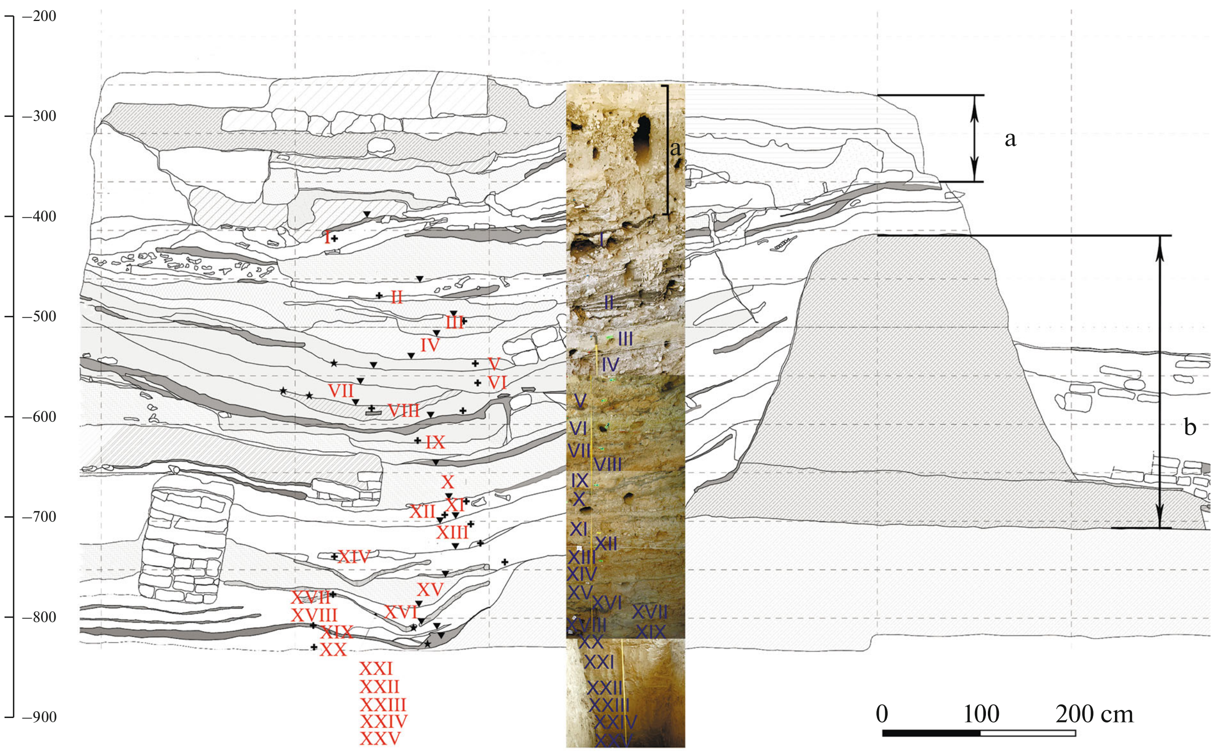


**Fig. 2.** The stratigraphic structure of the profile: (a) outer reinforcing layers of the later wall, composed of earthen material, (b) an early monolithic mudbrick wall. Drawing by D.A. Volkov and T.Yu. Cherezova.

## RESULTS

The total thickness of the cultural layer in the studied area is over 8 m (including earthen structures and their destruction products exposed on the daylight surface). We investigated the unconsolidated stratum of the cultural layer 661 cm thick, sealed under 2 m of the compact earthen material of the upper late part of the town wall (Fig. 2). The archaeological data and radiocarbon dating reliably date the sequence of the studied layers between the 7th and 10th centuries AD [14].

Twenty-five stratigraphic layers were identified and described in the studied section during the field work (I–XXV). During hand drilling at the bottom of the investigated trench in August 2018, the soil became water saturated at approximately 765 cm below the daylight surface (1005 cm from the “0” local reference mark). Given the loamy clayey texture, it could be argued that the lower horizons of the cultural layer in the studied stratigraphic column are now affected by groundwater.

In line with current classification concepts, the studied object falls into the category of technogenic surface formations and can be referred to the group of (paleo)urbiquasizems in the Classification of Soils of Russia [30], while in WRB system it belongs to Urbic Technosols (Archaic, Aridic, Calcaric, Loamic/Clayic, Salic) [56].

Figure 3 shows the distribution of analytical indicators for the study strata of the stratigraphic column: pH, $CaCO_3$, $CaSO_4$, $P_2O_5$, $C_{org}$, and electrical conductivity of water extract. Since the entire sediment column contains carbonates, the pH ranges around the carbonate equilibrium (8.3): from 7.4 (layer II) to 9.1 (layer XXV) (Fig. 3a). Note that layers containing large amounts of herbaceous detritus and/or charcoal-ash material have a pH just below the carbonate equilibrium but are in any case weakly alkaline. These layers show a slightly lower carbonate content. In the lower sediment unit, pH tends to be more alkaline. The carbonate content is high in all layers (Fig. 3b), ranging from 13% (layer XVIII) to 25% (layer XXIV).

The gypsum content varies from 4.3 (I) to 0.3% (XXV) (Fig. 3c), it has a wide variation all along the profile. The gypsum content is highest in the uppermost layers I and II showing 4.3 and 3.4%, respectively. Layers IV–V, VII–VIII, XI–XII, XV, and XVII contain up to 2–3% gypsum. The gypsum content in lower layers (XX, XXIV, and XXV) is below 1%.

The total phosphorus content is very high ranging from 0.2% (layer XXIV) to 12.8% (layer XV) (Fig. 3d). Organic carbon is unevenly distributed along the section: its maximum is in layer XVI with 3.1%, and the minimum of 0.2% is in layer XXV (Fig. 3e).

The value of specific electrical conductivity of the 1 : 5 aqueous extract ranges from 0.9 (layer XXIV) to 9.9 mS/cm (layer X) (Fig. 3f), suggesting that the degree of salinization of the layers varies from weakly to strongly saline [16]. Previously obtained indirect

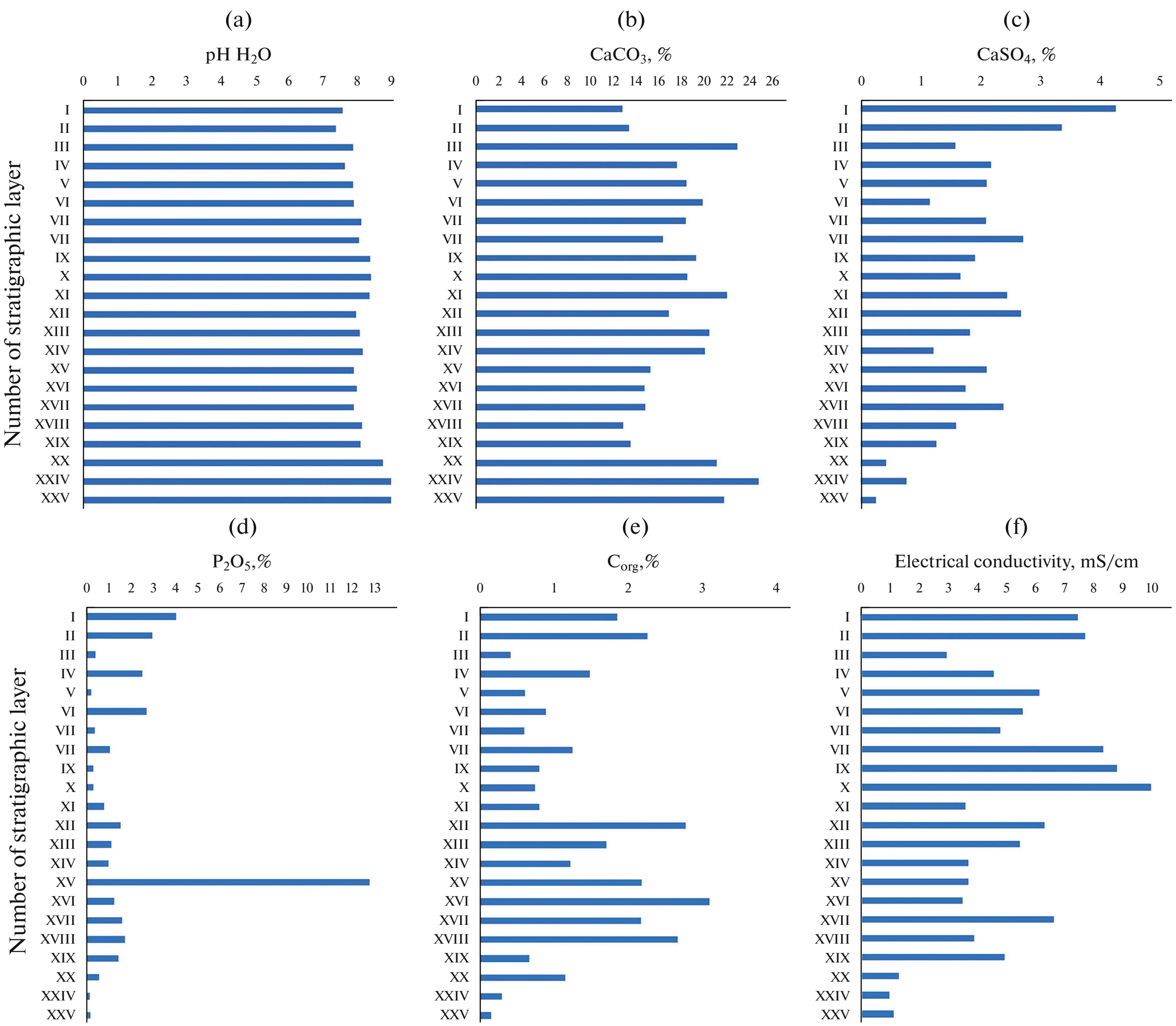


**Fig. 3.** Data of analytical studies of the stratigraphic layers: (a) pH $H_2O$; (b) $CaCO_3$, %; (c) gypsum, %; (d) $P_2O_5$ (total), %; (e) $C_{org}$, %; (f) specific electrical conductivity of the 1 : 5 water extract, mS/cm.

data (results of X-ray fluorescence bulk analysis and semiquantitative XRF-analysis in the course of electron microscopic studies) on the composition of easily soluble salts in the cultural layer of the Dzhankent settlement and adjacent reference soils suggest sodium-magnesium chloride-sulfate salinization [42].

Table S1 offers a brief macromorphological description of each layer. Table S2 contains a brief micromorphological description, and Table S3 demonstrates an experience-based semiquantitative assessment of the occurrence of microfeatures. Based on the assessment of the material composition of stratigraphic layers in the macromorphological study, the distribution of prevailing micromorphological features, as well as the content of organic carbon and total phosphorus, the studied stratigraphic column of the cultural layer was subdivided into three units: the upper unit, layers I–X; the middle, layers XI–XIX, and the lower, layers XX–XXV. Below, for each of the three units we offer generalized macro- and micromorphological descriptions, together with data on the varying total phosphorus (Fig. 3d) and organic carbon (Fig. 3e). The anthropogenic microfeatures are illustrated in Figs. 4a–4i, and the natural microfeatures, in Figs. 5a–5g.

**The upper unit** of layers (I–X) consists mostly of mineral and mineral-organic material. It is compact, medium and heavy loamy, has inclusions of plant detritus, large bone fragments, often well preserved (solid, undisintegrated), pottery, charcoals, and ash (Table S1). Isolated compacted mineral and mineral-organic microstrata, or blocks, often occur within a single layer (e.g., layers I, V, VI, and VIII); elsewhere, mineral and organic material is evenly distributed within a layer (layers IV and X). Layer IV contains concentra-

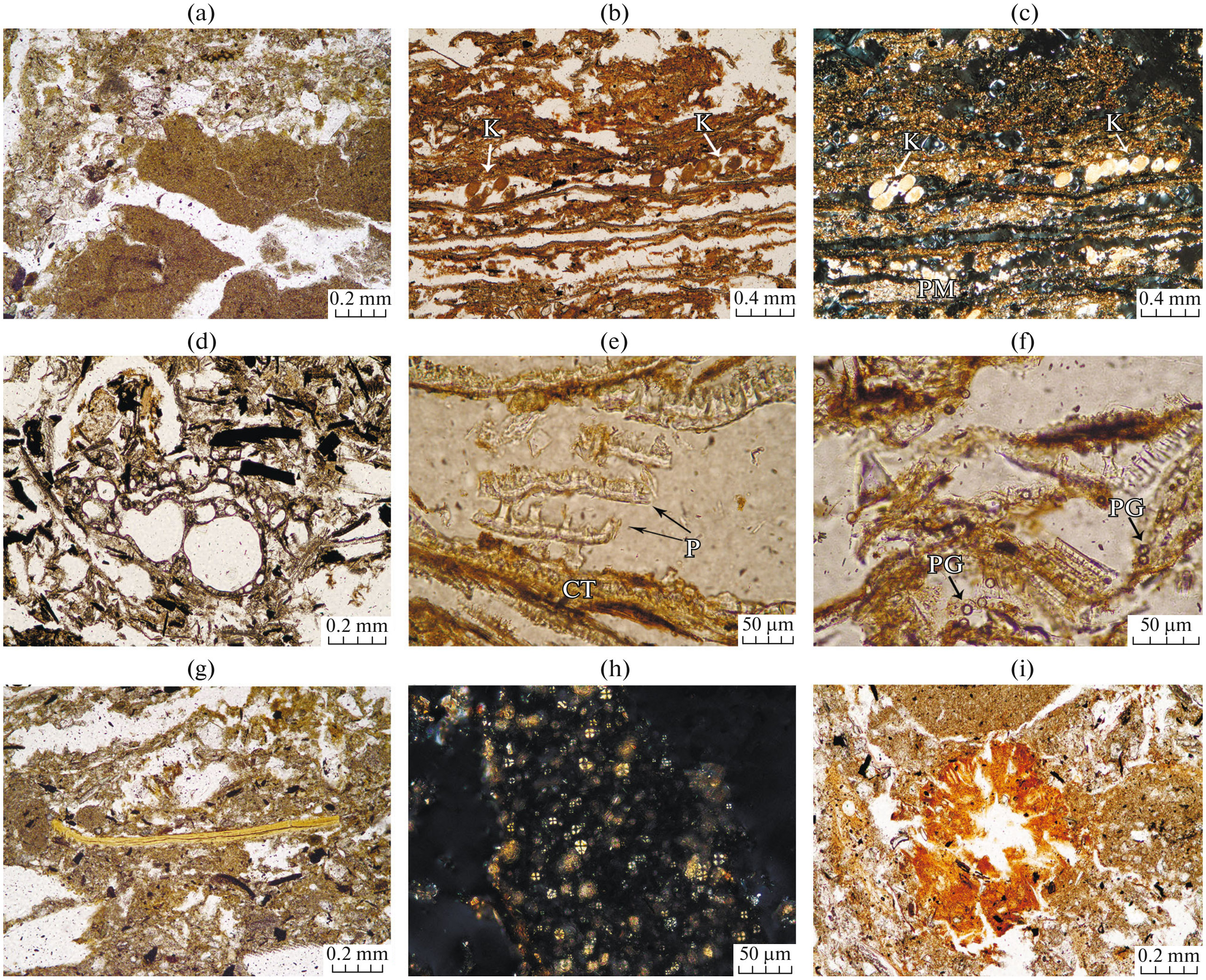


**Fig. 4.** Anthropogenic microfeatures: (a) carbonate-clay aggregates (layer X), PPL; (b) subparallel plant remains and coprolites (K) (layer XV), PPL; (c) subparallel plant remains encrusted with carbonates, coprolites (K) and pyrogenic micrite (PM) (layer XV), XPL; (d) vitrified mass with carbonized residue (layer XV), PPL; (e) phytoliths (P) in cereal tissues (CT) (layer XI), PPL; (f) pollen grains (PG) among plant tissues (layer XI), PPL; (g) a fish bone and fragments of charcoal (layer XIV), PPL; (h) aggregations of spherulites (layer XI), XPL; (i) a destroyed organophosphate nodule with charcoal fragments (layer XIII), PPL.

tions of soft powdery gypsum in the form of isometric or angular aggregates of irregular shape up to 3 mm in size. Since such formations, almost entirely consisting of finely dispersed gypsum, are often shaped as angular blocks and not observed in undisturbed soils, they can be considered anthropogenic inclusions.

At the microlevel (Tables S2 and S3), typical of this unit's layers is the presence of numerous angular blocky carbonate-clay aggregates (Fig. 4a). The fine material of such aggregates is often distinguished from the surrounding matrix micromass by darker color, dense groundmass and monic or porphyric C/F related distribution. Few bone fragments, often charred, are found in practically all layers. Apart from plant detritus, thin sections (layers II, IV, VII, and X) show phytoliths some of which have been identified as phytoliths of cereals (including cultivated cereals), rushes and reeds [63]. There are also microzones of organophosphate micromass (II, IV, and VIII) and grains of pollen (IV and VIII). Layers III and VIII show a traceable crumb microstructure of fine material (Fig. 5a). Almost all layers of this unit are characterized by numerous gypsum pedofeatures (Table S3): individual large lens-like crystals (Fig. 5b) (some with traces of dissolution) and their aggregates, and gypsum-salt infillings (VII and X, Fig. 5c). Layer VII contains isolated leaching pores left from fully dissolved large lens-shaped gypsum crystals (phantom pores). Various iron pedofeatures are found throughout this unit: coatings and hypocoatings (Fig. 5d), diffuse nodules, and ferruginated plant tissues (IV and VIII). Carbonate pedofeatures are represented by micrite coatings (V) and incrustations over plant tissues (IV and X).

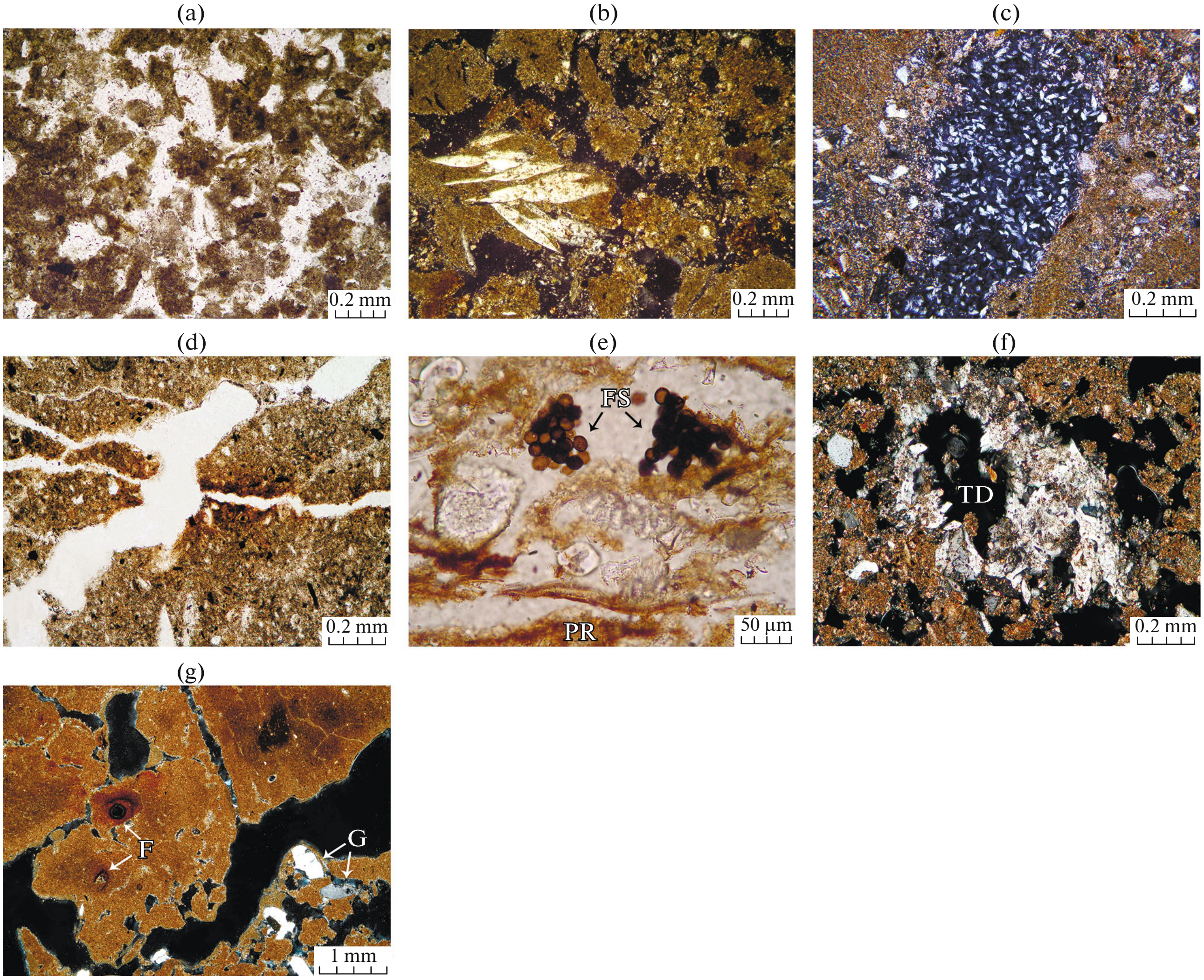


**Fig. 5.** Natural microfeatures: (a) crumb microstructure with impregnation by amorphous organic matter (layer III), PPL; (b) large lens-like gypsum crystals in aggregated carbonate clay material (layer III), XPL; (c) infilling with small rhomboid crystals of gypsum (layer IX), XPL; (d) iron-clay hypocoating in carbonate-clay material (layer I), PPL; (e) fungal spores (FS) among plant remains (PR) (layer XI), PPL; (f) gypsum-salt infilling with traces of dissolution (TS) (layer XIV), XPL; (g) ferrugination (F) of plant remains and gypsum crystals (G) in clay-carbonate material (layer XXIV), XPL.

Total phosphorus content varies significantly (Fig. 3d): it is below a fraction of a percentage point in the predominantly mineral and in the mineral-organic layers (layers III, V, VII, IX, and X), whereas in some mineral-organic layers (I, IV, VI, and VIII) and in layer II, rich in charcoals and ash, the content of total phosphorus is high and very high (1–4%). Organic carbon content is mostly low (Fig. 3e): it is less than 1% in the predominantly mineral layers, while in the mineral-organic layers, it is over 1%, exceeding 2% in layer II.

Layers of the **middle unit** (XI–XIX), loose, consist mostly of a mixture of organic and mineral materials and contain considerably more organic and organomineral substrates and pedofeatures than the unit above. Apart from the numerous charcoal-ash interlayers, this unit shows undisturbed pinkish interlayers of partially mineralized detritus up to 5–7 cm thick (XII, XV, and XVII) (Table S1). In many layers of this unit there are gypsum morphologies as angular, irregularly shaped aggregates up to 3 mm in size (XI–XII and XIV–XVI).

At the microlevel (Table S2), the charcoal-ash interlayers are represented by subparallel carbonized plant remains with micrite accumulations in between (Fig. 4b and Fig. 4c) and thin, long particles of grassy charcoal (layers XIII, XV, and XVII-XIX). The detritus in layers XV and XVIII shows isolated fragments of vitrified mass[2] (Fig. 4d). The presence of numerous chains of phytoliths of cultivated cereals, often near

[2] Vitrified mass is a glasslike mass formed by sintering of silicate minerals, quartz, and opal under high temperatures [59]. Morphologically, this microfeature is identified as dark mass, isotropic in crossed nicols, with rounded pores.

weakly decomposed plant tissues, was noted in all layers of this unit (Fig. 4e, Table S3). There are also numerous pollen grains (Fig. 4f) (XV, XVII, and XVIII), coprolites (Fig. 4b) (XV and XVIII), and sparse shells of diatoms with preserved valves (XVII). The bone material is fairly scant at the microlevel; however, layers XIII–XV and XVIII contain identified fragments of fish bones (Fig. 4g). Carbonate-clay aggregates are considerably fewer in this unit compared to the unit above, and they occur only in layers XIII–XV, and XVIII. The detritus in layer XI contains a large number of calcite spherulites (Fig. 4h) and fungal spores clusters (Fig. 5e). Organophosphate nodules were noted in layer XIII (Fig. 4i). All layers are abundant in carbonates encrusted over plant tissues (Fig. 4c), whereas ferruginated tissues are few (Table S3). Iron coatings and hypocoatings together with diffuse nodules are abundant in layers XIII and XIV, which are distinguished in this unit by the appearance of both gypsum-salt infillings (some with traces of dissolution) (Fig. 5f) and individual gypsum crystals in the carbonate-clay material.

The phosphorus content is moderately high at 0.8–1.8%, with the exception of layer XV (12.8%) (Fig. 3d). The organic carbon content in this unit is higher than in the unit above ranging from 0.7% (XIX) to 3.1% (XVI) (Fig. 3e).

Layers of the **lower unit** (XX–XXV) are very compact, medium- and heavy loamy, predominantly mineral with rare anthropogenic inclusions (Table S1). At the microlevel, layer XX (Tables S2 and S3) contains few charcoals, bone fragments, plant detritus, and phytoliths, with occasional coprolites and single pollen grains; crumb microstructure of fine material was noted. In contrast to the above units, there are multiple signs of iron redistribution here (Table S3): coatings and hypocoatings, diffuse nodules, and single ferruginated plant residues (Fig. 5g). Pedofeatures of gypsum and carbonates are few: we have observed carbonate incrustation over plant tissues in layer XX, and isolated gypsum crystals in the groundmass in layer XXIV (Fig. 5g).

The content of total phosphorus (Fig. 3d) and organic carbon (Fig. 3e) in this unit is low and decreasing with depth from 0.6 to 0.2% and from 0.7 to 0.3%, respectively.

## DISCUSSION

**Typification of diagnostically relevant features.** Morphogenetic studies of the cultural layer's stratigraphic column have made it possible to identify anthropogenic and natural, genetically significant microfeatures (mainly inclusions and pedofeatures) and diagnose the processes involved in their formation in the cultural layer (Table 1). Note that the subdivision of the entire set of features observed in the studied sequence of the cultural layer into anthropogenic and natural is somewhat notional. Almost all of the described features can be found both, in the soils formed without human participation and in the anthropogenically transformed soils and anthropogenic pedosediments. However, as we are dealing with cultural layers which were formed with human participation, we can assume that the abundance of such inclusions as carbonic particles and other features associated with burning (e.g., pyrogenic carbonates), various plant material (detritus, pollen), animal bones, phosphate pedofeatures, etc., is directly related here to the activity of medieval people. At the same time, the features associated with the redistribution and accumulation of carbonates, gypsum, and iron compounds are primarily due to natural conditions and processes. Nevertheless, some of these features, such as signs of redistribution and accumulation of iron in layers, or fragments of materials associated with earthen construction, could also have been produced with human involvement.

The **anthropogenic features** are believed to comprise inclusions and pedofeatures that appear in the layers as a direct result of human activity. These features are subdivided into two groups.

The ***first group*** comprises inclusions related to the input and redistribution of matter as a result of human settlement and economic activities. Human habitation is conducive to intense accumulation of various substrates depending on the type of anthropogenic activity: hearth and household detritus (food and human waste, bones, charcoal, and ash), handicraft and agricultural products (fragments of pottery, metallic slags, glass, manure, plant detritus associated with the storage of agricultural goods and forage for domestic animals), and building materials (bricks, plasters, remnants of earthen structures, and wood and other plant materials). Significantly, the studied layers in the profile are very rich in the quantity and diversity of anthropogenic microfeatures, suggesting a high anthropogenic impact in the area. The group of anthropogenic inclusions also comprises bone fragments, accumulations of calcite spherulites, and carbonate-clay aggregates. Plant detritus, phytoliths, pollen grains, and diatom shells were also included with the anthropogenic inclusions due to their abundance in the archeological context of the cultural layer and uneven distribution along the stratigraphic column.

*Plant detritus and phytoliths.* There is a large volume of plant detritus throughout the stratigraphic column, both at the macrolevel (Table S1) and at the microlevel (Table S3). Layers of the middle unit are distinctly enriched in detritus and phytoliths. At the microlevel, plant detritus occurs, as a rule, in two contexts: either in the mixture of mineral mass, bone fragments, and charcoals or as subparallel interlayers (Fig. 4b and Fig. 4c). In the former case, such a set of microfeatures may be pointing to the presence of hearth and domestic waste. In the latter case, together with subparallel strata of weakly decomposed plant remains, there are mass accumulations of various types of phytoliths, some of

**Table 1.** Interrelation between processes and microfeatures in the stratigraphic layers of the Dzhankent site

| Diagnostic features of processes | |
|---|---|
| Anthropogenic | Natural |
| *Processes of input and redistribution of matter* | *Processes of migration, sedimentation and recrystallization of matter* |
| Fragments of mammal and fish bones, plant detritus | Gypsum pedofeatures: large lens-like crystals and their intergrowths, gypsum and salt infillings |
| Phytoliths, including that of reeds, rushes, cultivated cereals, pollen grains, and diatoms | Carbonate pedofeatures: micrite coatings, incrustations over plant remains and coprolites |
| Calcite spherulites | Iron oxide pedofeatures: coatings and hypocoatings, diffuse nodules, and ferrugination over plant tissues |
| Carbonate-clay aggregates | |
| *Processes of transformation of matter* | |
| Pyrogenic features: accumulations of charcoal particles, pyrogenic carbonates, and fragments of vitrified silicate masses | Biogenic features: fungal spores, fruiting bodies of ascomycetes; coprolites |
| Organic and organophosphate pedofeatures | Crumb microstructure of fine material |

which are typical of the vegetative parts of cultivated cereals (Fig. 4e) [52, 63]. Their occurrence in unfragmented chains or even occasional bands (articulated phytoliths) seems to suggest that cereal crop residues were introduced directly into the layer and decomposed there in situ [71]. We have also found phytoliths typical of rushes and reeds.

*Pollen grains* (Fig. 4f) *and diatom shells* occur only within the middle unit whose layers are composed largely of organic matter (Table S3). We believe these biogenic inclusions were input there together with the abundant plant material by human activities.

*Bone fragments*. At the macrolevel, in particular in the upper unit, there is much large, well preserved bone debris, mostly of cattle and small ruminants [9] (Table S1). At the microlevel, although they have been observed along the entire column, bone fragments are few or sporadic (Table S3). Fish bone fragments were identified among other bone fragments (Fig. 4g). It would seem that conditions inside the soil do not favor disintegration of the bone material and inclusion of its microfragments into the total mass. Since large fragments of bones are not included in the studied samples, microfragments are few, and bone mineralization is suppressed, there is no definitive link that we can observe between the abundance of fossil microdetritus and bone microfragments in thin sections, and the values of organic carbon and phosphorus in the stratigraphic column.

*Calcite spherulites.* As noted above, calcite spherulites are numerous in layer XI (Fig. 4h, Table S3). Calcite spherulites are produced in the small intestine of animals by specific bacterial species. Large concentrations of spherulites would be observed in herbivorous animals [44, 45]. The presence of this microfeature is believed to be a marker of manure of herbivorous animals [64].

*Carbonate-clay aggregates* are found in many layers of the studied profile, and their especially large quantities are noted in the upper unit (Fig. 4a, Table S3). They are products of destruction of silicate-carbonate earthen building structures (walls and floors). Due to the scarcity of timber in Dzhankent, local carbonate-rich clay was used as a building material to make mudbricks, pakhsa, paste, and plaster covering floors and walls with layers of wet clay.

The ***second group*** of anthropogenic features includes the pedological features resulting from the transformation of human-introduced material by anthropogenic and/or processes in the soil. This group includes pyrogenic features, i.e., features related to the impact of fire or high temperatures: charcoals, accumulations of pyrogenic carbonates, and fragments of vitrified silicate masses.

*Pyrogenic carbonates* represented by micrite occur in clusters between charred or partially charred plant remains in layers IV, XV, and XVII–XVIII (Fig. 4c, Table S3). The same layers were described at the macrolevel as showing charcoal and ash interlayers (Table S1). In contrast to other carbonate forms, pyrogenic carbonates tend to accumulate in ash, and their presence is associated with the burning of the material. The source of carbonates in this case is the plant material. Plants contain calcium in the form of oxalates and oxides, which turn into calcium carbonates under temperatures over 200°C [3].

*Vitrified masses* (Fig. 4d) result from the sintering of high-silicon minerals such as quartz and opal at high temperatures [59]. The source of silica in these layers, in particular, may be phytoliths. The vitrified mass in the studied column was found in the middle unit, layers XV and XVIII (Table S3).

*Fragments of charcoal* (Fig. 4d) are the most common anthropogenic microfeature in the studied column, practically omnipresent throughout (Table S3).

This microfeature occurs, as a rule, together with plant remains. Not in all cases can we infer a direct impact of fire in the formation of any particular layer: what we need is not just the presence of large quantities of charcoals, but a whole set of microfeatures (charred bone fragments, pyrogenic carbonates, etc.).

The group of anthropogenic pedofeatures also includes *organic and organophosphate pedofeatures*: *impregnation (micromass)* (II, IV, VIII, XIX) and *nodules* (XIII) (Fig. 4i) (Table S3). Organophosphate nodules are formed through the transformation of phosphorus compounds input with organic matter of animal origin [57]. The amorphous organic impregnation was probably formed by the redistribution of organic matter within the layer [18].

**Natural features** in these layers are associated with migration, sedimentation and recrystallization of matter (concentrations of gypsum, carbonate, and iron oxides) and with processes of biological activity (fungal spores and coprolites).

Several microforms were noted among *gypsum pedofeatures* which differ in terms of the conditions of their genesis [18, 27, 65]:

(1) large, idiomorphic gypsum crystals within the aggregates, associated with slow crystallization from solutions in the water saturation zone (Fig. 5b);

(2) gypsum-salt infillings in pores, associated with the deposition from highly mineralized solutions under strong fluctuations of moistening and drying in arid conditions (Fig. 5c).

Gypsum pedofeatures at the microlevel occur in great quantities throughout the upper unit layers composed mostly of a loamy carbonate-silicate material (Table S3). These layers, as a rule, are rich in the fragments of destruction of earthen structures (walls and floors). Note that in this stratigraphic column there could have been two original sources of gypsum: anthropogenic, associated with building techniques, and natural, associated with soil solutions rich in sulfates and calcium. Making mudbricks and pastes, the builders could have added gypsum in the meal to make them stronger and water-resistant.

*Iron oxides pedofeatures*: diffuse mottles in the finely dispersed matter, coatings and hypocoatings in pores (Fig. 5d), and ferruginated plant residues are particularly common in some layers of the upper (III, VI, and VII) and middle units (XIII and XIV) (Table. S3). Such pedofeatures emerge in dynamic redox conditions [18, 24, 70]. It should be noted that iron pedofeatures in these layers are confined to zones composed mostly of mineral material with a high proportion of clay fine-dispersed matter, in particular, to morphologically distinguishable fragments of earthen building materials. This allows us to assume a certain contribution by the anthropogenic factor, namely, the use of water in the preparation of building raw materials and/or the erection of earthen structures.

In the lower unit (layers XX and XXIV), there are numerous hypocoatings in pores and diffuse nodules in the total mass; ferruginated plant remains are also found here (Fig. 5g). In this part of the section, iron pedofeatures are obviously related to the variable redox regime in the zone of periodic groundwater saturation [18, 67, 69, 70]. As noted above, during exploratory drilling in the studied column, before cutting into the lower layers of sediments, the ground becomes wet at a depth of 1005 cm from the "0" reference mark, i.e., at the level of stratigraphic layer XXIV.

*Carbonate pedofeatures* are represented by micrite coatings and incrustations over plant tissues (Fig. 4c). Carbonate incrustations are quite abundant in the layers of the middle unit (XI, XIII, XV, and XVII–XVIII), rich in plant detritus (Table S3). Both the presence of micritic coatings and carbonatization of plant remains are to be associated with migration, redistribution and sedimentation of carbonates from solutions in between carbonate-bearing layers [18].

Layers of the middle unit (XI and XV) have been shown to have various *biogenic features*: coprolites (Fig. 4b), fungal spores (Fig. 5e), and ascomycetes fruiting bodies (Table S3). The presence of such features suggests conditions in the soil favorable for this biota in the postanthropogenic period: temperature and humidity, availability of nutritious substrates, and absence of severe anthropogenic impact that would have been suppressed the biota (mechanical impacts or accumulation of toxic substances).

Some layers show fragmentary *crumb microstructure of fine material* (III, VIII, XIV, and XX) (Fig. 5a, Table S3). We assume that microstructure of this type in the studied layers can be related to salts. Salts affect the structural arrangement of fine material: they coagulate clay into small, rounded clusters, forming a loose packing of the material [18]. So, aggregation in layers III, XIV, and XX could have been caused by clay coagulation by carbonates, since these layers have a very high carbonate content (20–23%) (Fig. 3b). Microstructure of fine material in layer VIII is probably caused by easily soluble salts, as confirmed by the high value of electrical conductivity (8.3 mSm/cm) (Fig. 3f).

**A comparison of analytical and morphological data.** As noted above, all layers in the profile display a high content of *carbonates* (Fig. 3b). Overall, lower quantities of carbonates are typical of loose layers rich in biogenic and organic substrates: charcoals, plant detritus, and phytoliths. The higher carbonate content (around 20% and more) is typical of compact earthen layers and layers rich in the products of earthen materials demolition, as well as of the lower unit of heavy loamy-clayey sediments only slightly impacted by anthropogenic process and containing very few artifacts.

Occurrences of *gypsum* pedofeatures and inclusions (Fig. 3c) in layers correlate weakly with its composition determined by chemical analyses. Also, in some instances the reverse is true: layers with few gypsum pedofeatures are rich in $CaSO_4$. There is an interesting pattern in the ratio of carbonate and gypsum: the layers low in carbonates are high in $CaSO_4$, and vice versa. This may be partly due to exchange processes between clay-carbonate fine material and sulfate-saturated soil solutions, resulting in the formation of fine crystalline gypsum in the soil mass. In thin sections, its diagnostics can be difficult due to the small size of the crystals [18].

Variations in the *phosphorus* (Fig. 3d) and *organic carbon* (Fig. 3e) content in the stratigraphic column are expected to correlate with the apparent abundance of inclusions of plant detritus, bone fragments, charcoal, and organic and organophosphate pedofeatures. The contribution of each microfeature is difficult to assess on the scale of a cultural layer profile, so the entire set of carbon- and phosphorus-containing substrates should be assessed. Overall, the distribution of organic carbon and phosphorus correlates consistently with the proportion of organic substrates in the composition of layers: it is significantly higher in loose layers rich in herbaceous detritus, carbon ash, and bone material. The content of carbon and phosphorus in the middle unit of layers remains consistently high. Layer XV has the highest phosphorus content (12.8%); in this layer at the macro level an interlayer of organic detritus, possibly containing bone dust, has been described, while at the micro level fish bones are found.

Note that the general pattern of change in the analytical parameters along the stratigraphic column is fairly well traceable, both at the level of units of layers and in terms of differences between more mineral and more organic stratigraphic layers. However, attempts to correlate, for each individual layer, the compositional features observed in thin sections (abundance of carbonates, amount of gypsum, certain organic materials, phosphate pedofeatures, and bone remains) with analytical data obtained for this layer (content of carbonates, organic carbon, gypsum, and phosphorus) have often failed to reveal any regular relationship between morphological and analytical data. Possible reasons for such inconsistencies will be discussed further in the conclusions.

**Reconstruction of processes and conditions of stratigraphic units of the cultural layer formation.** The set of the obtained morphological and analytical characteristics of each layer and the identification of the predominant ones have helped to divide the entire studied column into three genetic-stratigraphic units.

The upper unit (I–X) consists of layers composed of compact loamy material which comes from products of destruction of carbonate earthen building structures (floors and walls), with charcoal and ash interlayers. Earthen fragments contain charcoal and plant detritus with phytoliths. In Central Asia, in addition to mudbricks, ash and lime-ash mixtures were widely used in construction since the ninth century. Ash was added between mudbricks as a waterproofing layer, and a lime-ash mixture was used as a binding agent [1]. The iron pedofeatures in earthen fragments may have been formed at the stage when clays were prepared for construction: when soaking for making mud bricks or when rolling wet clay to level the surface and/or make floors. A similar conclusion about the role of water-related technological processes (soaking loamy raw materials in water, subsequent laying and tamping of wet material) in the formation of pedofeatures involving iron oxides-hydroxides was made by the micromorphological study of construction techniques of earthen mound structures in the Southern Urals steppe zone [32].

The presence of numerous gypsum pedofeatures in the layers of this unit suggests dramatic fluctuations in moistening and prolonged desiccation of gypsum- or sulfur-bearing layers under arid conditions. These conditions are evidenced by numerous signs of gypsum leaching: erosion of individual large crystals and crystals within infillings up to complete dissolution of gypsum resulting in the formation of leaching pores shaped as lenticular crystals.

The layers of the middle unit (XI–XIX), by the combination of their predominant features, belong to the economic layers rich in hearth and food wastes. They represent living surfaces: alternation of more or less loose organomineral and organic strata and rolled-on clay floors, which from time to time sealed the garbage accumulated on the surface. Here we note a high content of phosphorus and organic carbon, as well as the greatest variety and abundance of anthropogenic features: bones, organophosphate pedofeatures and calcite spherulites, and carbonate-clay aggregates. A large number of charcoal-ash interlayers are present in this unit. Described in this unit are interlayers composed of morphologically preserved herbaceous detritus with the inclusion of numerous phytoliths of cultivated cereals, reeds, and rushes. Obviously, such a combination of plants reconstructed from the phytoliths cannot be a component of the same phytocenosis. It is possible that the varied plant material belonging to coarse cereals (crop residues of cultivated cereals and reeds) and sedges (rushes) was used as matting or the covering material for floors and roofs, or in the construction of light roofs. Hay and straw are still widely used in this region in the construction of household and residential structures: barns, fences, livestock pens, and light-frame dwellings. The abundance of phytoliths of hydrophilic plants and phytoliths of the vegetative part of cultivated cereals can be taken as indirect evidence of more favorable, less water-deficient, conditions in the formation of this sediment unit compared to the present-day conditions. The abundance and diversity of anthropogenic inclusions and pedofeatures point to a strong anthropogenic impact on the area during the formation of the middle layer unit. So, the formation of this unit must have taken place during the peak of human habitation in the area.

The layers of the lower unit (XX–XXV) are composed of very dense, highly carbonate, medium and heavy loamy and clayey alluvial-deltaic sediments with few anthropogenic features. The anthropogenic impact associated with this unit of layers was minimal. Numerous iron pedofeatures indicate a variable redox regime in the groundwater fluctuation zone.

## CONCLUSIONS

The morphogenetic studies have been used to describe macro- and micromorphological and physicochemical properties and their variation in the full stratigraphic column of the cultural layer in the early medieval town of Dzhankent, which had the developed infrastructure of an agrarian and craft center, built in the traditions of earthen architecture in the desert landscape of the Syr Dar'ya paleodelta. Our studies made it possible to inventory the natural and anthropogenic features and their corresponding processes in the cultural layer formation, to trace changes in the set of these features along the stratigraphic column of the cultural layer, and to substantiate the stratigraphic division of the cultural layer into units, or groups of layers, corresponding to the three main stages in its formation.

The general characteristics of the composition and properties, and the set of microfeatures of the cultural layer are shaped not only by anthropogenic processes, but also by natural factors and processes, in particular by zonal features, as well as geochemical and local hydrological conditions. Above all, natural conditions and processes predicate the presence of the signs of accumulation and redistribution of carbonates and gypsum, associated with arid climate, together with iron oxides-hydroxides, associated with a dynamic redox regime in the capillary fringe of groundwater. However, the presence, diversity and special features of carbonate, gypsum, and iron pedofeatures morphology and their distribution in the stratigraphic column are regulated not only by natural, but partly by anthropogenic processes.

Overall, our research shows that there is a great potential in studying the cultural layers of archaeological sites with the application of hierarchical morphology in combination with traditional methods for studying chemical composition and physical and chemical properties. It has been shown that the physicochemical and chemical characteristics of the cultural layer and their changes within the studied stratigraphic column agree well with the morphosubstantive characteristics at the level of distinctions between more mineral and more organic layers, as well as at the scale of the units of layers. The morphosubstantive features of the layers described at the micromorphological level such as their degree of calcification, content of organic materials, gypsum, and phosphorus-containing microfeatures (bones, phosphate pedofeatures) often correlate poorly with the analytical data on the content of carbonates, organic carbon, gypsum, and phosphorus. The main reason for these discrepancies lies, primarily, in the extremely high heterogeneity of stratigraphic layers both lateral (planigraphic heterogeneity) and in depth (microstratigraphic heterogeneity). In addition, micromorphological studies without the use of additional research methods (e.g., micro-XRF studies of thin sections) only allow for qualitative or semiquantitative information on the composition. Such information is always subjective to a degree. In addition, it also depends on the size of the components, the resolution of the microscope, and certain difficulties in diagnosing one or another feature. In particular, routine studies using thin sections cannot assess the fine silt and clay fraction components even semiquantitatively, and often cannot even identify them.

All these challenges in the analysis and interpretation can be partially or completely overcome with the help of the techniques that are being actively explored today to study undisturbed stratigraphic columns of the cultural layer, the so-called geoblocks, by various destructive and nondestructive methods [39]. With this approach, we can address microstratigraphic and microplanigraphic heterogeneity by obtaining continuous data series for undisturbed monoliths (different types of high-resolution scanning, micro-XRF mapping, etc.) and/or parallel sampling in the same context (microlayer, lateral microheterogeneity) for all types of analyses used in the study.

## SUPPLEMENTARY INFORMATION

The online version contains supplementary material available at https://doi.org/10.1134/S1064229324604141.

Table S1. Macromorphological description of the stratigraphic layers of the Dzhankent site.

Table S2. Micromorphological description of the stratigraphic layers of the Dzhankent site.

Table S3. Experience-based semiquantitative assessment of microfeatures occurrence in the stratigraphic layers of the Dzhankent site.

## ACKNOWLEDGMENTS

The authors are grateful to A.E. Ivanova, Candidate of Biological Sciences, Leading Researcher at the Department of Soil Science, Moscow State University, for help in identifying the elements of ascomycete structure; Dr. A.A. Golyeva, Senior Researcher, Department of Geography and Evolution of Soils, Institute of Geography, Russian Academy of Sciences, for identification of phytoliths in slides; and Dr. A.V. Panin, Corresponding Member of the Russian Academy of Sciences, Head of the Department of Quaternary Paleogeography, Institute of Geography, Russian Academy of Sciences, for consultative assistance in writing this paper.

Micromorphological studies were carried out at the Laboratory of Radiocarbon Dating and Electron Microscopy of the Institute of Geography, Russian Academy of Sciences.

## FUNDING

The research by J.O. Karpova and M.A. Bronnikova was carried out as part of the government assignment to the Institute of Geography of the Russian Academy of Sciences, FMWS-2024-0010. The study by M.P Lebedeva was conducted as part of the research topic of the Dokuchaev Soil Science Institute, No. 0439-2022-0013.3.

Field studies, preparation of thin sections, and micromorphological and analytical studies were funded by DFG project 389351859.

## ETHICS APPROVAL AND CONSENT TO PARTICIPATE

This work does not contain any studies involving human and animal subjects.

## CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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*Translated by G. Ermakova*