

Phase composition and microstructural heterogeneity of ancient ceramics from east Kazakhstan: raman spectroscopy, x-ray microtomography and scanning electron microscopy

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Abstract. This study presents the results of a comprehensive investigation of ceramic fragments from the Shagantogay archaeological site (Makanchi District, Abay region, Republic of Kazakhstan) using modern combined analytical techniques: Raman spectroscopy, X-ray microtomography, and scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS). Raman spectroscopy revealed a heterogeneous mineral composition of the ceramics, comprising quartz, anatase, feldspars, and carbonate phases. X-ray tomographic examination demonstrated significant differences in the internal structure of the ceramic samples, ranging from dense and homogeneous to highly porous with inclusions of organic origin. SEM-EDS data confirmed the variability of elemental composition, which correlates with the identified mineral phases. Applying these techniques together allowed the ceramic material to be studied at several levels — phase composition, internal structure, and elemental distribution — with minimal impact on the artifacts. The data obtained describe the composition of the clay matrix and the structural features of the Shagantogay archaeological site and may be useful for further comparative work on ceramic assemblages in Eastern Kazakhstan.

Keywords: Ancient pottery; microstructure of ceramics; X-ray microtomography; Scanning electron microscopy (SEM); Raman spectroscopy; Shagantogay archaeological site.

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Introduction

The Shagantogay archaeological site is located 14 km northwest of the village of Karabulak in the Makanchi District of the Abay Region of the Republic of Kazakhstan (at the foot of the Barlyk Mountains, in the Alakol Basin). The site is a multilayered complex comprising various types of archaeological structures. A distinctive feature of this site is its location within a sand dune massif characterized by high aeolian activity. Winds shift sand masses, causing the site's surface to deflate and accumulate. This leads to the periodic exposure of archaeological features and cultural layers, followed by their subsequent re-covering with sand. As a result, the site's modern microtopography is significantly altered, making it difficult to determine the original positions of artifacts. Therefore, systematic monitoring, regular photographic recording, and the use of high-precision spatial documentation methods are required. During the 2023 field season, the Tarbagatai Archaeological Expedition (led by R.S. Zhumatayev) conducted reconnaissance work at this archaeological complex. During the survey, excavated material was collected, consisting of fragments of ceramic artifacts dating back to various historical periods. A preliminary analysis of the ceramic material recovered from the Shagantogay archaeological site suggests a high population density in this area. The local population's main economic activities included livestock breeding, agriculture, and fishing. Furthermore, the typological and technological diversity of the ceramic assemblage indicates extensive trade, economic, and cultural contacts between the site's population and border regions. Structural features may provide much-needed data for developing historical hypotheses about the methods and techniques of pottery production, clarifying ideas about trade networks, and cultural interactions between different civilizations [1].

For structural studies, samples of ceramic material from the archaeological finds from Shagantogay were selected and systematically examined using scanning electron microscopy (SEM), X-ray microtomography, and Raman spectroscopy. SEM allows for high-resolution imaging of ceramic surfaces, revealing microstructural features, particle morphology, and textural heterogeneities in the ceramic materials [2]. When combined with energy-dispersive X-ray spectroscopy (EDS), SEM provides localized data on elemental composition, facilitating the identification of raw materials, additives, and surface treatments. Furthermore, SEM analysis of grain boundaries and microstructural features can provide important information on production methods and firing conditions [3].

X-ray microtomography allows visualization of internal cracks, layers, and other physical features inaccessible using conventional techniques, making it particularly valuable for studying fragile objects that cannot be subjected to destructive analysis [4–6]. This technique has become indispensable for assessing production methods and the state of preservation of archaeological ceramics [7]. Raman spectroscopy offers unique possibilities for the chemical characterization of ceramic materials. The method's sensitivity to both crystalline and amorphous phases makes it particularly useful for the study of ancient ceramic materials [8].

This paper presents a comprehensive quantitative correlation between phase composition, microstructural porosity and elemental heterogeneity in Shagantogay ceramics using Raman spectroscopy, X-ray microtomography and SEM-EDS.

Materials and methods

Sample Descriptions

For structural analysis, we selected six ceramic fragments from the Shagantogay complex. The ceramic samples were designated S-1, S-2, S-3, S-4, S-5, and S-6 (Figure 1).

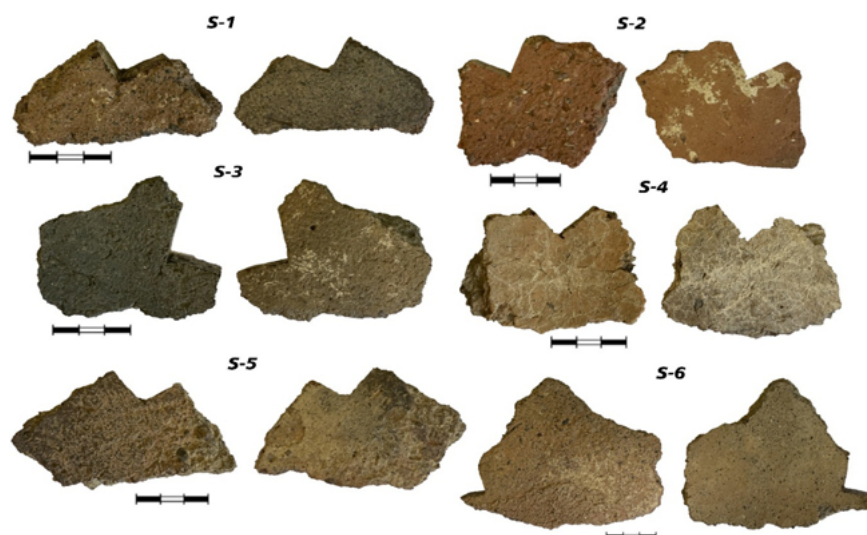


Figure 1. The photos from two sides of the ceramic fragments. The scale bar is shown 2 cm.

Raman Spectroscopy

Raman spectroscopy was used for the preliminary characterization of the phase composition of the ceramic samples. The measurements were carried out using a Confotec Duo confocal Raman spectrometer (SOL Instruments GmbH, Augsburg, Germany) equipped with an optical microscope. Spectral excitation was provided by laser operating at a wavelength of 532 nm. Raman spectra were acquired under confocal conditions using a $\times 20$ microscope objective. Measurements were performed at several representative points on the surface of the investigated samples in the spectral range of 50–2000 cm^{-1} , with an acquisition time of 2–4 min per point. Phase identification was achieved by comparing the obtained Raman features with reference spectra reported in the literature [9].

X-Ray microtomography

X-ray microtomography, performed on an IMAX (PRODIS Compact) system [10], was employed to investigate the internal structure of the ceramic fragments. This technique enables the acquisition of three-dimensional images of an object without causing its destruction, allowing for the detection of internal defects (voids, inclusions, and cracks), the examination of fastener geometry, and the identification of pathways for the ingress of corrosion and contaminants into the archaeological objects under study. The X-ray microtomograph operates with a maximum spatial resolution of up to 1 μm . For the analysis of the ceramic artifacts, the X-ray tube parameters were set to 70 kV and 50 μA . The dimensions of the reconstructed voxels were $80 \times 60 \times 60 \mu\text{m}^3$. Visualization, processing, and quantitative analysis of the resulting three-dimensional data were performed using VGStudio MAX 2.2 software (Volume Graphics).

Scanning electron microscopy (SEM).

Fragments of ceramic samples were examined using a TESCAN VEGA 3 scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectroscopy (EDS) detector [11]. The SEM-EDS experiments were conducted at the Laboratory of Radiation Biology, Joint Institute for Nuclear Research (JINR), Dubna, Russia. The instrument enables elemental composition analysis by means of energy dispersive X-ray spectrometers capable of detecting elements with atomic numbers of 5 and above, beginning with boron. The method allows elemental analysis of the ceramic body, identifying such elements as aluminium (Al), silicon (Si), calcium (Ca), sodium (Na), iron (Fe), magnesium (Mg), potassium (K), and others [12]. For SEM-EDS analysis, a small fragment was cut from each ceramic sample to a diameter of 10 mm using a diamond saw. The technical parameters of the SEM-EDS system are provided (Table 1).

Table 1. Technical characteristics of the TESCAN VEGA 3 microscope during measurement

Name	Spectrum
Processing time	5 s
Accelerating voltage	15.00 kV
Working distance	15.0 mm
Take-off angle (degrees)	33.7°
Azimuth (degrees)	0.0°
Number of channels	2048
Energy range (keV)	20 keV
Energy per channel (eV)	10.0 eV
Detector ID	26
Detector type	X-Act
Window type	SATW
Sum peak correction	Successfully completed
Primary detector serial number	75042
Dwell time	5 µs

Results and discussion

Raman spectroscopy

Raman spectroscopy enables characterisation of the mineral composition of inclusions on the surface of ceramic material from the Shagantogay historical complex. Representative Raman spectra corresponding to different mineral components identified in the ceramic samples under study (Figure 2). Comparative spectral analysis allowed identification of the following mineral phases: quartz (SiO_2), anatase (TiO_2), feldspars and minor components including carbon, calcite, and hematite (Fe_2O_3) (Table 2). Quartz, detected in all samples examined, displays characteristic peaks at 127, 205, 357 and 464 cm^{-1} [13]. Feldspars were identified by characteristic bands at 290, 478, and 513 cm^{-1} in samples S-3, S-4, S-5, and S-6 [14]. The presence of anatase (TiO_2) is confirmed by a well-defined peak at 146 cm^{-1} , with a notably stronger signal observed in samples S-1, S-2, S-3, and S-5.

Of particular interest is the detection of hematite (Fe_2O_3) in sample S-6, evidenced by characteristic bands at 226, 295, 412, 612, and 1322 cm^{-1} [15], indicating oxidising firing conditions for this ceramic fragment [16]. Carbon or soot phases identified in samples S-3, S-4, S-5 and S-6 are likely associated with the use of organic temper materials – such as straw, plant stems, or grog – incorporated during ceramic manufacture [17,18]. These organic additives are consumed during the firing of ceramic vessels.

The firing temperature of ceramic vessels can be estimated from the positions of the Raman D and G bands of the carbon doublet at ~ 1340 and ~ 1570 cm^{-1} , respectively, using the HD/HG intensity ratio [17]. Sample S-3 (HD/HG ≈ 0.65) corresponds to a relatively low firing temperature (~ 700 °C), whereas samples S-5 and S-6 (HD/HG ≈ 0.80 – 0.81) indicate intermediate firing temperatures (~ 800 – 850 °C). The highest ratio was recorded for sample S-4 (HD/HG ≈ 0.86), suggesting a firing temperature approaching 900 °C (Figure 3). It should be noted that the detection of anatase in the majority of samples indicates a firing temperature below ~ 850 °C [19]. It can therefore be inferred that the ceramic fragments under study were fired at moderate temperatures in the range of ~ 700 – 900 °C. However, the localised presence of anatase, hematite, or carbon phases may reflect minor variations in firing conditions or ceramic manufacturing practices across individual vessels.

Table 2. Distribution of mineral phases in ceramic samples based on Raman spectroscopy results. The symbol “*” indicates reliable phase identification.

Sample	Phase composition					
	Quartz	Anatase	Feldspar	Carbon	Calcite	Hematite
S-1	*	*				
S-2	*	*				
S-3	*	*	*	*		
S-4	*		*	*	*	
S-5	*	*	*	*		
S-6	*	*	*	*		*

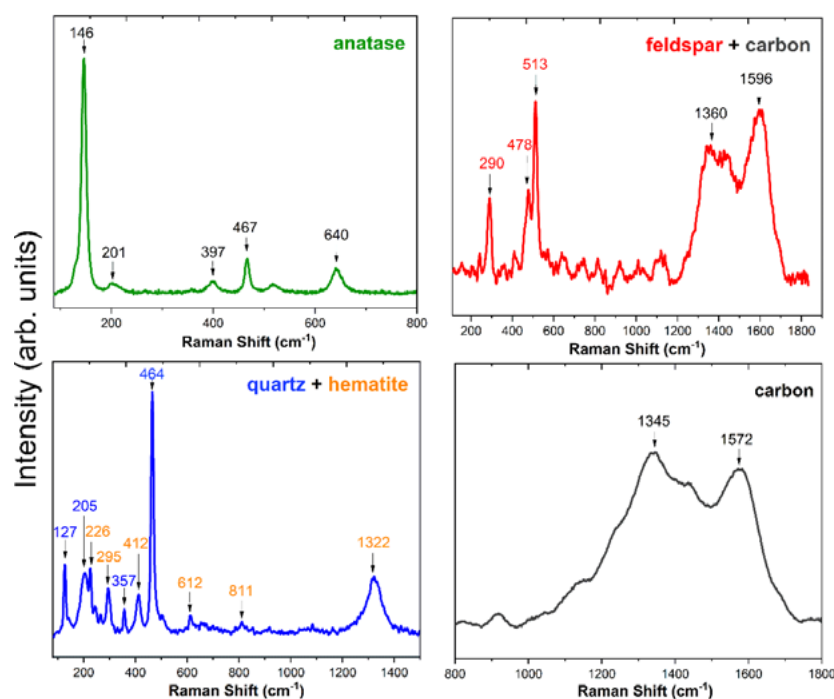


Figure 2. Characteristic Raman spectra of mineral additives identified in the investigated ceramic fragments. The positions of the Raman lines and the identified mineral components are presented.

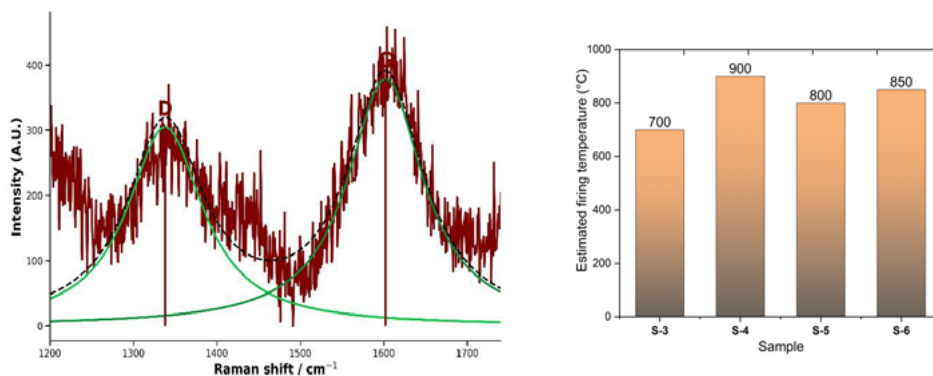


Figure 3. An example of the analysis of the positions of the carbon HD/HG doublet in a Raman spectrum. Estimation of the annealing temperatures of the investigated ceramic material—recovered from archaeological excavations at Shagantogay—based on the ratio of the HD/HG Raman peaks.

X-ray microtomography

The X-ray microtomography experiment was performed under the following conditions: an X-ray tube voltage of 70–80 kV, a current of 50 μ A, an image pixel size of 35–45 μ m, and a voxel size after reconstruction of 35 $\times$ 35 μ m². A 150 μ m-thick copper filter was used to suppress the low-energy component of the spectrum. Tomographic data were acquired in the step-by-step sample rotation mode, with up to 900 projections recorded at an angular step of 0.4°, averaging 5 frames per projection, and an exposure time of 100–400 μ s.

The spatial distribution of internal pores and mineral inclusions in six archaeological ceramic samples from the Shagantogay complex was investigated using X-ray tomography. Three-dimensional (3D) models of the studied fragments, reconstructed from the tomography data (Figure 4).

The reconstructed tomographic data of the studied ceramics was used to segment the internal components (Figure 4) [20]. The tomographic reconstruction of sample S-1 reveals a medium proportion of internal inclusions (~11.25%, Table 3) and a moderately porous internal structure without large elongated voids, which indicates relatively stable firing conditions. Sample S-2 demonstrates the highest proportion of internal inclusions among all the studied ceramics samples, approximately 17.96%, which indicates an extremely heterogeneous internal structure (Table 3). The reconstructed data of sample S-3 revealed spiral voids and a large volume of internal pores, and the calculated relative volume of inclusions reaches values of up to ~15.86%. The complex shape of the internal voids in sample S-3 indicates the possible use of organic additives (leaners), such as chopped straw or grass, which completely burn out during the firing of ceramic products. Organic and mineral impurities compensated for the natural shrinkage of clay during drying and firing, which reduced the likelihood of deformation and cracking of the products [21]. Tomographic reconstruction revealed a moderate volume fraction of internal inclusions (~9.50%), dispersed pores and several internal cracks in the ceramic matrix. Compared to samples S-2 and S-3, the structure of this sample appears relatively denser and less heterogeneous, indicating a comparatively higher firing intensity. Sample S-5 demonstrates a relatively compact internal structure with a moderate volume fraction of internal inclusions (~7.56%) and only slight development of large pores in tomographic sections (Figure 4, b).

The ceramic matrix appears relatively homogeneous, although it still contains small dispersed voids and minor structural heterogeneities. On the other hand, ceramic sample S-6 is distinguished by a more homogeneous structure and the lowest volume of inclusions - 6.52%.

The observed technological variability is consistent with previously described characteristics of the steppe ceramic traditions of Eastern Kazakhstan [18,22], where moderate firing temperatures, heterogeneous mineral inclusions and organic additives were widely used in small-scale ceramic production systems.

Table 3. Calculated data on the volume of components of the studied ceramic fragments from computed tomography analysis.

Sample	Total Volume, voxels	Impurities Volume, voxels	Proportion of Impurities, %
S-1	205 039 305	23 070 464	11.25%
S-2	250 416 949	44 982 776	17.96%
S-3	186 385 988	29 568 613	15.86%
S-4	172 203 072	16 356 915	9.50%
S-5	304 843 484	23 063 917	7.56%
S-6	210 874 051	13 740 851	6.52%

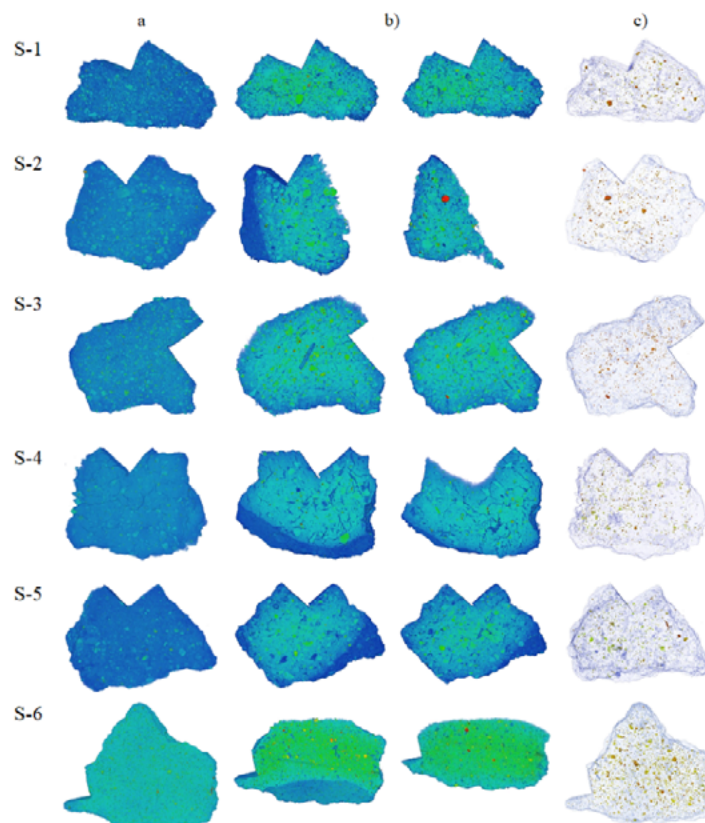


Figure 4. 3D models (a) created after tomographic reconstruction and several longitudinal slices (c) of studied ceramic fragments. Rainbow coloring shows the degree of X-ray absorption from low (blue) to high (red). The highlighted internal inclusions inside ceramic fragment volumes (b).

Scanning electron microscopy (SEM)

Scanning electron microscopy combined with energy-dispersive spectroscopy was used to study the morphology, microstructure, and chemical composition of ceramic samples S-1, S-2, S-3, S-4, S-5, and S-6. The analysis was performed at multiple surface points (from 16 to 55 measurements depending on the sample), which made it possible to evaluate both the average values of the elemental composition and its spatial variability. The average values of the percentage content of elements for all measured points (table 4). In all studied samples, the main elements are oxygen (O), silicon (Si), and aluminum (Al), indicating the dominance of the aluminosilicate matrix characteristic of clay materials. The content of these elements varies within relatively narrow limits, indicating the overall stability of the basic composition and the homogeneity of the clay base.

Element mass fraction distribution diagrams for all studied samples (Figure 5). This type of visualization allows for the simultaneous evaluation of median values, interquartile ranges, ranges of variation, and outliers reflecting extreme values. In almost all samples, the elements O, Si, and Al are characterized by narrow interquartile ranges, stable median values, and minimal outliers. This confirms that they form the main matrix of the material. In contrast, the elements Ca, Fe, and Ti exhibit a wide range of values, extensive variations, and numerous outliers. This pattern indicates the presence of localized mineral inclusions that differ in composition from the main matrix. Extreme values for individual elements are associated with the electron beam hitting individual mineral grains, process additives, or impurities.

Table 4. Average values of elemental composition (wt%) of ceramic samples according to SEM-EDS data

Element	S-1 (n = 28)		S-2 (n = 48)		S-3 (n = 16)	
	Average, wt.%	±SD	Average, wt.%	±SD	Average, wt.%	±SD
O	58.97	4.92	55.02	6.50	56.23	6.18
Na	1.37	1.42	1.42	1.17	1.25	1.47
Mg	2.05	1.44	1.85	1.65	1.39	1.25
Al	6.89	1.99	8.93	3.47	8.35	3.91
Si	18.20	4.76	20.36	6.93	18.68	7.41
P	0.92	0.83	0.42	0.31	0.35	0.11
S	0.45	0.30	0.32	0.12	—	—
K	1.91	1.24	2.26	1.25	2.29	1.14
Ca	5.64	5.49	3.86	7.86	2.87	4.63
Ti	0.30	0.09	1.70	6.74	3.22	7.50
Mn	0.43	0.10	1.04	0.03	1.14	0.73
Fe	3.53	1.92	5.61	7.36	7.11	9.41
Element	S-4 (n = 23)		S-5 (n = 54)		S-6 (n = 27)	
	Average, wt.%	±SD	Average, wt.%	±SD	Average, wt.%	±SD
O	58.77	5.77	55.60	3.91	60.42	3.77
Na	0.64	0.26	2.42	2.44	1.31	1.86
Mg	1.67	0.59	1.10	0.49	1.33	0.44
Al	9.48	10.43	9.09	3.36	7.86	2.49
Si	16.32	7.09	23.29	6.26	20.31	3.99
P	3.16	5.71	0.26	0.08	2.10	2.45
S	0.38	0.13	0.45	0.17	—	—
K	1.68	0.66	2.11	1.12	1.67	0.85
Ca	7.61	8.94	4.38	6.68	2.78	2.78
Ti	1.19	3.08	0.53	0.85	0.58	1.08
Mn	0.37	0.00	—	—	0.44	0.00
Fe	3.85	5.06	2.89	1.78	3.17	2.40

Note: “—” means the element was not detected in this sample; SD means standard deviation.

In sample S-1, the base is an aluminosilicate matrix (O ~45–65%, Si ~10–23%, Al ~5–10%) with a stable presence of Na, K, and Mg reflecting the admixture of feldspar material. A characteristic feature of the sample is the variability of the Ca content: in most points 3–8%, but in some points it increases to ~15–22% in combination with S ~0.5–1.2%, which indicates the presence of calcium sulfate phases (gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ or anhydrite CaSO_4), associated with post-depositional alteration or with the use of lime lean agent. Iron (Fe) is present at a moderate level (1–8%) without significant outliers. Sample S-2 is characterized by the most homogeneous aluminosilicate composition among the studied samples: median values of Si (~21%) and Al (~9%) are stable, and the interquartile range for the major elements is minimal.

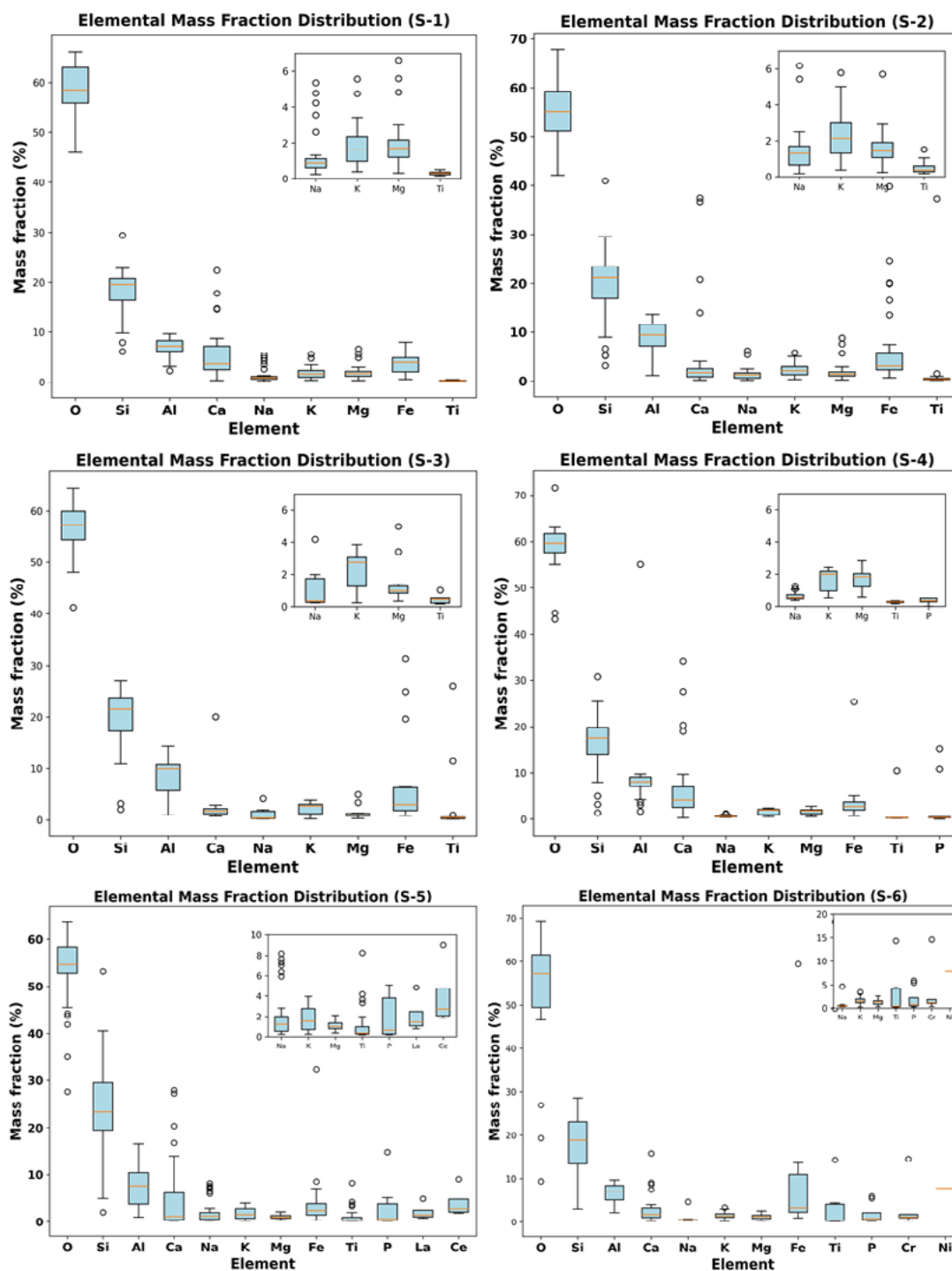


Figure 5. Scatter of elemental mass fractions (wt.%) in the investigated ceramic samples, based on SEM-EDS data, presented as a boxplot. Outliers are indicated by open white circles. The inset panels illustrate the distribution of elements with low concentrations (0–10 wt.%).

Anomalous elemental concentrations were recorded at isolated points in sample S-2. One spectrum showed a high Fe content (~40%) accompanied by low Si and Al concentrations, which is consistent with the presence of an Fe-rich oxide phase, possibly corresponding to magnetite (Fe_3O_4) or hematite (Fe_2O_3). Another spectrum showed a high Ti content (~37%), with an elemental composition close to that expected for a TiO_2 -rich phase, possibly rutile. The Fe- and Ti-rich inclusions are interpreted as accessory mineral components. The identification of hematite

and anatase by Raman spectroscopy provides independent mineralogical evidence for the occurrence of Fe- and Ti-bearing phases in the studied ceramics. The high Ca content ($\sim 37.46\%$) combined with elevated O ($\sim 50\%$) and the absence of P and negligible S identifies this spectrum as a calcite (CaCO_3) inclusion, most likely derived from limestone temper or a calcareous clay raw material.

Sample S-3 contains individual microinclusions enriched in Fe and Ti (Figure 5). The most interesting measurement is Spectrum 81 (Figure 6), with a high concentration of both elements in equal proportions: Ti $\sim 26\%$, Fe $\sim 25\%$, while Si $\sim 3\%$ and Al $\sim 1\%$ are low. This elemental composition is consistent with an Fe-Ti-rich oxide phase and is possibly compatible with ilmenite (FeTiO_3). However, because SEM-EDS alone does not provide definitive crystallographic phase identification, this assignment should be considered tentative. The next measurement point (Figure 6), with a lower Ti content of $\sim 11.4\%$ and a high Fe content of $\sim 31.3\%$, is consistent with an Fe-rich oxide phase, possibly hematite (Fe_2O_3) with a minor Ti contribution (no Si). The presence of hematite is independently supported by the Raman spectroscopy results for the studied ceramic assemblage. At one point in ceramic sample S-4, the elemental composition is characterized by the dominance of aluminum (55.08%) with a high oxygen content (43.21%) and minor amounts of silicon (1.40%) and calcium (0.30%). This elemental composition is consistent with an Al-rich oxide phase, with a composition approaching Al_2O_3 . The same sample also exhibits localized calcium content maxima (up to $\sim 35\%$), which correlate with the presence of carbonate or calcium silicate phases. Furthermore, at individual points in samples S-4 and S-5, areas enriched in calcium and phosphorus (up to $\sim 15\%$) were identified, indicating the presence of microphases interpreted as calcium phosphate compounds similar in composition to apatite.

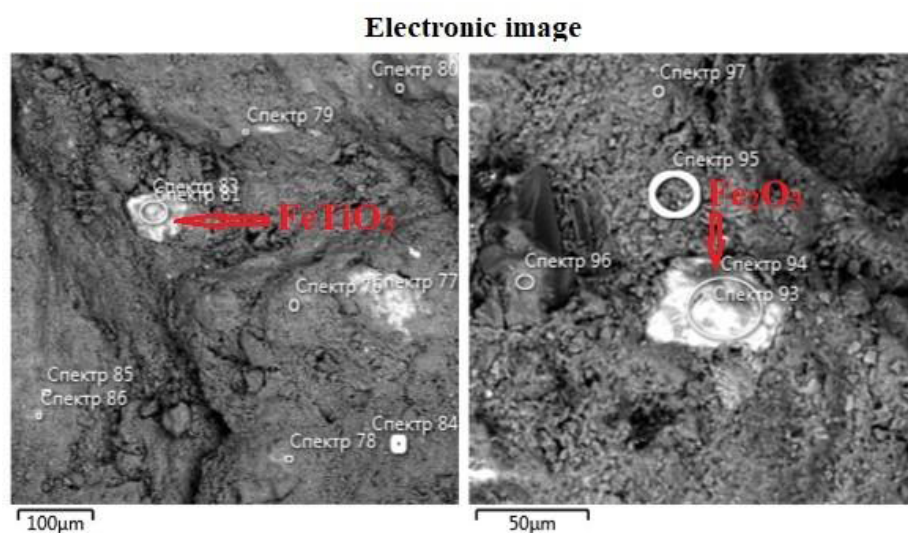


Figure 6. SEM images for sample S-3. (Спектр-Spectrum).

S-5 is also characterized by a pronounced multicomponent structure and a high degree of chemical heterogeneity. A distinctive feature of this sample is the presence of the rare earth elements La ($\sim 0.7\text{--}4.8\%$) and Ce ($\sim 1.8\text{--}9.0\%$), the distribution of which is discrete and associated with individual mineral inclusions. Minor elements with low relative concentrations: Na, K, Mg, P, Cr, Ni, and others. Of particular interest is sample S-6, which recorded values of Cr $\approx 14.6\%$ and Ni $\approx 7.8\%$, which may indicate the presence of rare minerals (e.g., spinels), or that these minerals are part of the contaminants (Figure 5). Sample S-6 exhibits the highest degree of chemical heterogeneity of all the samples studied. The matrix is also composed of an aluminosilicate (O $\sim 50\text{--}65\%$, Si $\sim 15\text{--}30\%$, Al $\sim 5\text{--}14\%$), but the element distribution is characterized by extreme values. In particular, the Fe content varies from $\sim 1\%$ to $\sim 60\%$, and Ti from a fraction of a percent to $\sim 70\%$, indicating the presence of individual iron-bearing phases and titanium-bearing minerals (Figure 5, S-6).

It should also be taken into account that local analytical effects (the influence of surface topography and the electron beam interaction volume) are possible during SEM-EDS analysis, which can introduce certain uncertainties in determining the concentrations of individual elements.

As an example, the authors conducted EDS mapping of sample S-1 (Figure 7). The dominant elements O, Si, and Al exhibit a relatively uniform distribution over the entire surface of the fragment, while Ca and Fe are concentrated in localized areas, indicating the heterogeneous nature of the inclusions.

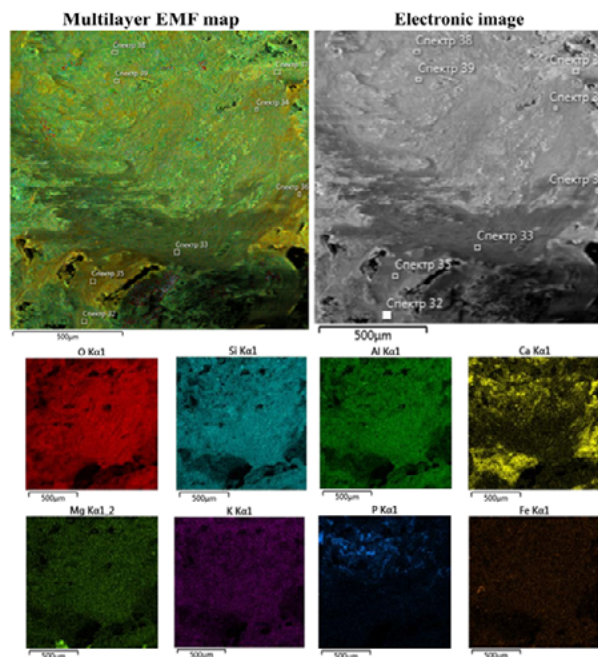


Figure 7. Microphotographs of ceramic sample S-1. The multilayer map displays the distribution of these elements, where color intensity corresponds to the mass fraction of the element in various regions of the sample (Спектр-Spectrum).

A detailed analysis of eight selected points (Spectra 32–39, Table 5) allows us to clarify the nature of this heterogeneity. Six of the eight points (Spectra 32–34, 37–39) reflect the typical composition of an aluminosilicate matrix: O ~55–59%, Si ~15–20%, Al ~7–8%, K ~2–3%, Fe ~4–7%, which corresponds to the clayey base of the sample.

Table 5. Results of elemental analysis of sample S-1 from different selected points of the microscopic image (Figure 7).

Element	32		33		34		35	
	Wt.,%	σ ,%	Wt.,%	σ ,%	Wt.,%	σ ,%	Wt.,%	σ ,%
O	57.9	0.2	59.1	0.2	58.5	0.2	65.3	0.2
Na	0.3	0.1	0.7	0.1	0.8	0.1		
Mg	6.6	0.1	1.9	0.1	1.7	0.1	0.8	0.0
Al	5.6	0.1	7.9	0.1	7.3	0.1	2.3	0.1
Si	16.3	0.1	19.1	0.1	19.5	0.1	6.1	0.1
P			0.3	0.0	0.9	0.0	0.2	0.0
S	0.2	0.0	0.3	0.0	0.2	0.0	0.9	0.0
K	1.4	0.1	2.2	0.1	2.3	0.1	0.7	0.0
Ca	4.1	0.1	3.4	0.1	4.3	0.1	22.3	0.2
Ti	0.3	0.1	0.3	0.1	0.3	0.1		
Mn								
Fe	7.2	0.2	4.9	0.2	4.2	0.1	1.5	0.1

Element	36		37		38		39	
	Wt.,%	σ ,%	Wt.,%	σ ,%	Wt.,%	σ ,%	Wt.,%	σ ,%
O	66.0	0.2	57.5	0.2	55.4	0.2	57.3	0.2
Na	0.3	0.1	0.9	0.1	1.1	0.1	0.8	0.1
Mg	0.9	0.1	2.1	0.1	2.1	0.1	1.6	0.1
Al	3.1	0.1	8.3	0.1	8.3	0.1	7.0	0.1
Si	7.9	0.1	19.2	0.1	19.5	0.1	15.4	0.1
P	0.2	0.0	0.4	0.0	1.3	0.1	3.2	0.1
S	1.1	0.0	0.3	0.0	0.3	0.0	0.3	0.0
K	0.8	0.1	2.7	0.1	3.4	0.1	2.0	0.1
Ca	17.8	0.1	3.1	0.1	3.7	0.1	7.8	0.1
Ti			0.4	0.1	0.3	0.1	0.4	0.1
Mn			0.3	0.1				
Fe	1.9	0.1	4.8	0.2	4.5	0.2	4.2	0.2

High concentrations of calcium, especially at some points (Spectra 35, 36), as well as its weak correlation with other elements ($r < 0.3$) according to the Pearson correlation coefficient (Table 6), may indicate the presence of limestone mineral admixtures (gypsum or anhydrite). It is noteworthy that Ti is not detected in spectra 35 and 36, whereas it is consistently present at levels of ~ 0.3 – 0.4 wt% in the other locations, further confirming the distinct mineralogical nature of these regions. Phosphorus (P) is recorded at low concentrations (0.2–1.3%) at several locations, reaching a maximum in spectrum 39 (3.2%), which is consistent with the presence of dispersed phosphate micro inclusions also found in other samples of the complex.

Table 6. A correlation matrix is presented showing the relationship between the chemical elements of ceramic samples. Pearson correlation coefficient (r) values range from -1 to 1, with positive values indicating a direct relationship and negative values indicating an inverse relationship.

Element	O	Na	Mg	Al	Si	K	Ca	Ti	Fe
O	1.00	0.66	-0.89	-0.56	-0.64	-0.98	0.72	0.80	-0.19
Na	0.66	1.00	-0.92	0.03	0.06	-0.72	0.04	0.96	0.48
Mg	-0.89	-0.92	1.00	0.20	0.24	0.92	-0.34	-0.96	-0.25
Al	-0.56	0.03	0.20	1.00	0.84	0.53	-0.94	-0.23	0.83
Si	-0.64	0.06	0.24	0.84	1.00	0.52	-0.95	-0.15	0.86
Ti	0.80	0.96	-0.96	-0.23	-0.15	-	-	1.00	-

The strongest positive correlation was demonstrated by Ti and Na ($r = 0.96$), which may indicate a similar geochemical origin. Meanwhile, Si and Ca showed a strong negative correlation ($r = -0.95$), indicating possible differences in phase composition.

Statistical analysis of elemental composition data

The obtained SEM-EDS data and their comprehensive analysis in combination with multivariate Principal Component Analysis (PCA and k-means clustering) statistics allow for a detailed statistical analysis of the results [23]. Before applying PCA, the data are often centered (mean subtracted) and normalized so that the features have the same scale (mean 0, variance 1). Normalization:

(1)

where $\bar{x}$ – average value for each feature, σ – standard deviation.

Principal component analysis (PCA) of SEM-EDS point spectra ($n = 196$) obtained from six ceramic fragments (Figure 8) showed that the first (PC1, 24.5%) and second (PC2, 19.6%) principal components jointly explained

44.1% of the total variance in the chemical composition. The remaining 55.9% of the variance is distributed among higher-order principal components and reflects additional compositional variability that is not represented in the two-dimensional PC1–PC2 projection. Therefore, the PC1–PC2 biplot is used primarily to visualize the major compositional trends and the principal compositional groups, rather than to represent the complete variance of the dataset. The interpretation of the compositional groups was additionally supported by k-means clustering ($k = 3$). K-means clustering ($k = 3$; value optimized using the elbow method and silhouette coefficient) allowed us to distinguish three distinct geochemical groups. Cluster 2, which covers the majority of the spectra, represents an aluminosilicate clay matrix (Si–Al–K), characteristic of all samples and indicative of a single clay source. Cluster 1 reflects inclusions enriched in Ca and P, corresponding to carbonate and phosphate phases; Cluster 3, in turn, corresponds to oxide minerals of Fe and Ti (ilmenite, iron oxides), present as accessory inclusions. The distribution pattern of individual samples on the PCA biplot in particular, the overlap of samples S-1, S-2, and S-5 within Cluster 2 – indicates technological similarities in the selection of the source material; sample S-4, however, demonstrates a more pronounced affinity with Cluster 1, which is consistent with the elevated Ca content recorded in the average chemical composition data for this sample.

All ceramic samples (Table 7) share a similar aluminosilicate clay matrix (Cluster 2, 78–90% of the spectra), which suggests the use of compositionally similar raw materials. However, the present SEM-EDS, Raman spectroscopy, and micro-CT data are insufficient to conclusively establish a common clay source. The main clay matrix is characterized by a stable positive correlation between Si and Al, confirming the preservation of the aluminosilicate structure. Differences between the samples reflect varying proportions of secondary mineral inclusions: sample S-4 has a higher content of carbonate and phosphate inclusions (Cluster 1: 17.4%), while samples S-3 and S-6 are characterized by a higher proportion of Fe–Ti oxide grains (Cluster 3: 12.5% and 11.1%, respectively). No Fe–Ti phases were detected in samples S-1 and S-4. The Fe–Ti-enriched cluster identified by PCA corresponds well to the hematite and anatase phases detected by Raman spectroscopy, particularly in samples S-3 and S-6, indicating oxidative annealing conditions and the presence of accessory iron and titanium minerals. Similarly, the Ca–P-enriched cluster correlates with calcite-containing regions observed in Raman spectra and calcium-rich inclusions detected by SEM-EDS, particularly in sample S-4.

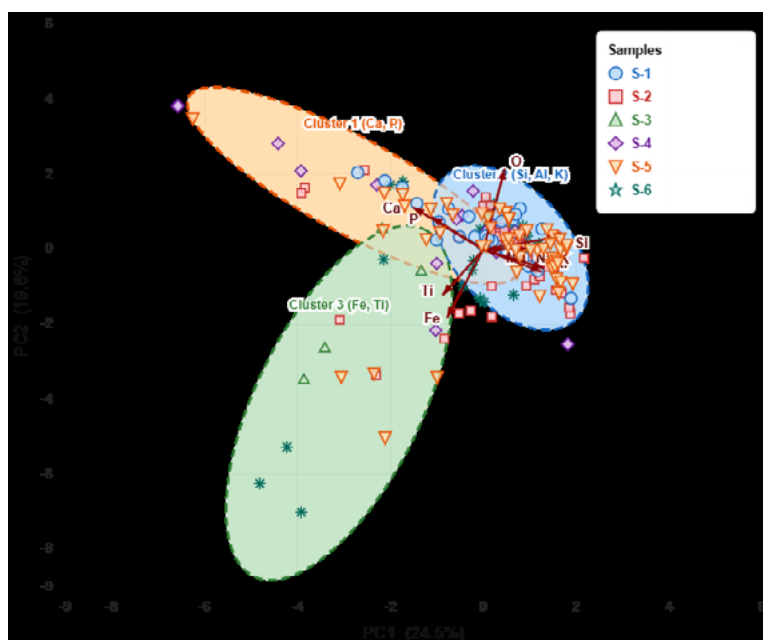


Figure 8. Principal Component Analysis (PCA) biplot of SEM-EDS point spectra ($n = 196$) from six archaeological ceramic fragments (S-1–S-6). Each marker represents a single EDS spectrum; marker shape denotes the sample. Dashed ellipses represent 95% confidence regions of three compositional groups identified by k-means clustering ($k = 3$): Cluster 1 – Ca, P-enriched spectra (carbonate and phosphate inclusions); Cluster 2 – Si, Al, K-dominant aluminosilicate matrix; Cluster 3 – Fe, Ti-enriched spectra (oxide mineral inclusions). Cluster 1 – Ca, P-enriched spectra (carbonate and phosphate inclusions); Cluster 2 – Si, Al, K-dominant aluminosilicate matrix; Cluster 3 – Fe, Ti-enriched spectra (oxide mineral inclusions).

Table 7. Distribution of SEM-EDS spectra across three k-means compositional clusters for each ceramic sample (S-1, S-2, S-3, S-4, S-5, S-6), expressed as percentage of total spectra per sample.

Sample	n total	Cluster 1 (Ca, P)	Cluster 2 (Si, Al, K)	Cluster 3 (Fe, Ti)
S-1	28	14.3% (4)	85.7% (24)	-
S-2	48	6.2% (3)	89.6% (43)	4.2% (2)
S-3	16	6.2% (1)	81.2% (13)	12.5% (2)
S-4	23	17.4% (4)	82.6% (19)	-
S-5	54	13% (7)	79.6% (43)	7.4% (4)
S-6	27	11.1% (3)	77.8% (21)	11.1% (3)

Conclusion

This paper presents a comprehensive study of Shagantogay ceramics using Raman spectroscopy, X-ray microtomography, SEM-EDS elemental mapping, and multivariate statistical analysis to reconstruct ceramic production technology in Eastern Kazakhstan. The analytical methods show good agreement. Raman spectroscopy identified quartz, feldspar, anatase, hematite, calcite, and carbonaceous phases, while SEM-EDS confirmed Si–Al, Ti, Fe, and Ca enrichment. Tomography revealed differences in porosity and internal structural heterogeneity. The preservation of calcite in sample S-4 indicates firing below the CaCO_3 decomposition threshold, whereas hematite in sample S-6 suggests oxidizing conditions and higher firing intensity. Raman carbon band intensity ratios (HD/HG) indicate firing temperatures of approximately 700–900°C. Samples with higher carbonate or organic inclusion contents show greater pore connectivity and heterogeneity, while denser aluminosilicate matrices correspond to the dominant Si–Al–K PCA cluster. PCA and k-means clustering confirm that mineralogical differences are reflected in ceramic composition. Overall, the results indicate that technological factors, including temper addition, homogenization, and

temperature control, play an important role in determining the structural variability of the ceramics. The observed similarity of the aluminosilicate matrices may suggest the use of compositionally similar raw materials; however, the available data do not allow a definitive assessment of differences or similarities in clay provenance.

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Zhomartova A.Zh.: Conceptualization, Methodology, Investigation, Writing – Original Draft; **Zhumatayev R.S.:** Investigation, Visualization, Resources; **Nazarov K.M.:** Investigation; **Kenessarín M.:** Investigation; **Mukhametuly B.:** Project administration, Supervision, Funding acquisition; **Saprykin E.A.:** Investigation; **Myrzabekova E.:** Investigation; **Kichanov S.E.:** Conceptualization, Methodology, Writing – review & editing;

Conflict of Interest

The authors declare that they have no conflict of interest related to the publication of this article.

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Шығыс Қазақстаннан табылған ежелгі керамиканың фазалық құрамы және микроқұрылымдық гетерогенділігі: раман спектроскопиясы, рентгендік микротомография және сканирлеуші электрондық микроскопия

Аңдатпа. Бұл зерттеу Шағантоғай археологиялық ескерткішінен (Мақаншы ауданы, Абай облысы, Қазақстан Республикасы) табылған керамика сынықтарын заманауи бұзбайтын талдау әдістерін — Раман спектроскопиясын, рентгендік микротомографияны және энергия-дисперсиялық рентгендік спектроскопиясы бар сканирлеуші электрондық микроскопияны (SEM-EDS) қолдана отырып, кешенді зерттеу нәтижелерін ұсынады. Раман спектроскопиясы керамиканың минералдық құрамының гетерогенді екенін көрсетті, оған кварц, анатаз, дала шпаттары және карбонат фазалары кіреді. Рентгендік томографиялық зерттеу керамика үлгілерінің ішкі құрылымында айтарлықтай айырмашылықтардың бар екенін көрсетті — тығыз әрі біртекті құрылымнан бастап, органикалық тектес қосындылары бар жоғары кеуекті құрылымға дейін. SEM-EDS деректері анықталған минералдық фазалармен сәйкес келетін элементтік құрамның вариативтілігін растады. Бұл әдістерді кешенді түрде қолдану керамикалық материалды бірнеше деңгейде — фазалық құрамы, ішкі құрылысы және элементтердің таралуы бойынша — артефактілерге барынша аз әсер ете отырып зерттеуге мүмкіндік берді. Алынған деректер Шағантоғай археологиялық ескерткішінің керамикасының сазды негізінің құрамы мен құрылымдық ерекшеліктерін сипаттайды және Шығыс Қазақстанның керамикалық кешендерімен одан әрі салыстырмалы жұмыс жүргізуде пайдалы болуы мүмкін.

Түйін сөздер: Ежелгі керамика; керамиканың микроқұрылымы; рентгендік микротомография; сканирлеуші электрондық микроскопия (SEM); раман спектроскопиясы; Шағантоғай археологиялық ескерткіші

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Фазовый состав и микроструктурная неоднородность древней керамики из восточного Казахстана: рамановская спектроскопия, рентгеновская микротомография и сканирующая электронная микроскопия

Аннотация. Данное исследование представляет результаты комплексного изучения фрагментов керамики с археологического памятника Шағантоғай (Мақаншинский район, область Абай, Республика Казахстан) с использованием современных неразрушающих аналитических методов: рамановской спектроскопии, рентгеновской микротомографии и сканирующей электронной микроскопии с энергодисперсионной рентгеновской спектроскопией (СЭМ-ЭДС). Рамановская спектроскопия выявила гетерогенный минеральный состав керамики, включающий кварц, анатаз, полевые шпаты и карбонатные фазы. Рентгенотомографическое исследование продемонстрировало значительные различия во внутренней структуре образцов керамики — от плотной и однородной до высокопористой с включениями органического происхождения. Данные СЭМ-ЭДС подтвердили вариативность элементного состава, которая коррелирует с выявленными минеральными фазами. Применение этих методов в комплексе позволило изучить керамический материал на нескольких уровнях — фазовый состав, внутреннее строение и распределение элементов — при минимальном воздействии на артефакты. Полученные данные описывают состав глинистой основы и структурные особенности керамики археологического памятника Шағантоғай и могут быть полезны для дальнейшей сравнительной работы с керамическими комплексами Восточного Казахстана.

Ключевые слова: Древняя керамика; микроструктура керамики; рентгеновская микротомография; сканирующая электронная микроскопия (СЭМ); рамановская спектроскопия; археологический памятник Шағантоғай

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