



Main Characteristics of the Raw Material Used for Obtaining Materials for the Restoration of Historical Monuments

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ABSTRACT: This article used modern methods of physical and chemical analysis to study the properties of raw materials. In our studies, the chemical, mineralogical composition, physical and mechanical properties of ancient Khiva's used historical monuments, ceramic bricks, and masonry mortars were studied. After studying these properties, we selected new formulations to develop alternative materials. We have studied the possibilities of using modifying additives, as a result of which an identical brick will be obtained in terms of color characteristics. During the research, the chemical, mineralogical and granulometric compositions of historical bricks, masonry mortars, and clay raw materials were determined, and their ceramic-technological and thermophysical properties were studied. The chemical composition of raw materials was studied by X-ray phase studies using non-traditional modern physical and chemical methods, the mineralogical composition of raw materials was determined by scanning (scanning) microscopy, and the granulometric composition of raw materials was determined by the dry method and sedimentation analysis. The raw materials for the development of materials for the restoration of architectural monuments were the loess-like rocks of the Suzanlinskoye deposit, waste from sugar production – defecation formed at the JV JSC “Khorezm Shakar” and amorphous silica. Loess-like rocks are predominantly light brown or gray in color, consisting of minerals that form a complex structure. According to the data obtained, a comprehensive study of loess-like rocks is of great scientific and practical importance, since more than 70% of the territory of the Republic of Uzbekistan is made up of loess-like and loess-like rocks.

KEYWORDS: Amorphous silica, Defecation, Granulometric composition, Loess loam, Restoration ceramic brick.

INTRODUCTION

The wall material of the historical objects of ancient Khiva is ceramic brick obtained by firing loess-like loams, which were carried out in a reducing environment in homemade khumbuz ovens, where the heat source was the heat released from the combustion of various solid fuels. The resulting material retained its external dimensions and color for many years. But in recent years, under the influence of natural factors, the processes of destruction of historical objects, including the regions of the Aral Sea world heritage, have been activated [1]. In this connection, the production of restorative materials with high mechanical strength and chemical resistance has become relevant. Indicator structures with a low XRD coefficient ($R_p = 8-11\%$) are typical for models with defects. Here, additional structurally defective and previously studied models are discussed [1].

According to the information presented in [2], attempts to improve the properties of limestone compositions as solutions in construction have been made since ancient times. It is recommended to add polysaccharides, proteins (animal glues and casein), and fatty acids (olive oil) to this source to improve them. Six types of mortar mixtures have been developed and characterized. The mechanical compressive strength, water resistance, degree of carbonization, porosity, structure and mineral composition have been studied and new mortar mixtures with additional advantages have been proposed on their basis. All compounds are compatible with traditional building materials, so it is scientifically proven that they can be used to restore historical and modern architecture used in brick walls.

The problems of the increasing use of lime-based mixtures for the repair and restoration of historical monuments and structures are also identified. During the study, the ratio of binder and filler (B/Ag), filler properties, and porosity were investigated. Mixtures prepared with lime, various types of aggregates, and B/Ag ratios from 1:1 to 1:5 by volume were tested. Measurements of



the compressive strength, as well as X-ray diffraction (XRD) and thermal studies of the samples, were carried out after 3, 7, 28, 91, 182, and 365 days. It was found that after 365 days of hardening (compared to hardening after 28 days), the strength of the mortar mixtures increased. The inclusion of a large amount of binder and mixture in the mixture also increases the strength of the clay stone. The resource studies the relationship between mechanical properties and porosity structure. However, it has been reported that increased porosity with an excess of binder reduces strength [3].

In another source [4], Chinese scientists prepared a mixture based on the calcined ginger nut and natural hydraulic lime in order to study the strength improvement mechanism of traditional lime-based binders using ground clay plaster and their mechanical strength systematically studied the hydration kinetics and microstructural properties. The results showed that calcined ginger oil was well compatible with clay soil and compared to lime-based mixtures, calcined ginger oil-based mixtures showed higher compressive strength, lower thermal conductivity, denser microstructure, and similar pore size distribution compared to with source material, clay plaster in later stages.

Another source [5] studied the resistance of mixed clays to salt solutions. The movement of soluble salts through the layer of the mixture is analyzed and it is established that this process varies depending on the porosity of the mixture.

Also in another source, scientists have shown that different results are obtained when using lime, hydraulic lime, or Portland cement as a binder in mixed clays and that the chemical reaction depends on the macro- and microporous structure of the mixtures [5, 6].

In other words, it was proposed to use polymers and alkoxysilanes in composite clay [7], but the authors considered this method to be ineffective in preserving ancient historical monuments.

This work was also done by C.T. Oguchi and others also found it ineffective [8]. In another source, the authors studied the addition of foam and rock wool to the composition of mixed clay, and according to their research, the addition of mineral wool to the composition of mixed clay increases the mechanical effect on bending, has a positive effect on cold resistance reduces expansion from heat and shrinkage from cold, and in mixed clays with the addition of polystyrene, the opposite was observed [9].

In the agricultural sector of the Aral Sea region, there is a huge amount of rice processing waste containing amorphous silica in its composition. In recent years, great attention has been paid to the use of amorphous silica in the process of obtaining ceramic materials [10].

A number of requirements are imposed on the restoration materials used from ceramic bricks. In addition to mechanical and chemical properties, it must fully match in.

EXPERIMENTAL TECHNIQUE

Our methodological studies have studied the chemical, mineralogical composition, physical and mechanical properties of the used historical monuments of ancient Khiva, which used ceramic bricks and masonry mortars. After studying these properties, we selected new compositions for the development of alternative materials.

In particular, the chemical, mineralogical, and granulometric compositions of historical bricks, masonry mortars, and clay raw materials were determined, and their ceramic-technological and thermal properties were studied.

The study used modern methods of physical and chemical analysis to study the properties of raw materials. The phase composition of the samples was studied using a Rigaku SmartLab 3 X-ray diffractometer. In the measurement process, CuK α radiation and the luminescence suppression mode (XRF) were used. The sample volume is not less than 0.02 cm³, and the angular resolution of reflections is up to 0.01°. Scanning speed 10°/min. The radiographs were interpreted using Crystallographica Search-Match v. 2.0.3.1 Oxford Cryosystems.

The dispersed composition of the samples of dried sediments was determined by the method of laser radiation scattering using a SALD-2201 Laser Diffraction Particle Size Analyzer (Shimadzu, Japan). The mineralogical composition of the raw material was determined by scanning (raster) microscopy TESCAN VEGA 3 SBH.

The technological properties of the raw materials used in the study were carried out in accordance with the state standards GOST 21216-2014 [11], introduced in the CIS countries, and the results obtained were compared with the data given in the source [13].

The granulometric composition of raw materials was determined according to GOST 21216.2-93, GOST 9169-2021, and OST 21-78-88 [12] by the dry method and the method of sedimentation analysis. In this case, the raw material was mixed with a



certain amount of water and the method of fractionation in a gravitational field was used, the main device was a laser particle analyzer: Shimadzu, SALD-7500 nanodevice [14].

The determination of the plasticity of raw materials was carried out by the Atterberg method, in accordance with the requirements of GOST 21216.1-93 [15], introduced in the CIS countries. In this case, the value of the elasticity index of the sample prepared from raw materials was determined from the difference between the flow surface limit and the flat surface moisture limit [16].

To determine the sensitivity of raw materials to drying, traditional methods and norms of GOST 530-95 were used [17].

Obtained from the raw materials of nearby quarries around ancient Khiva, in terms of its coloristic indicators, it does not correspond to its predecessor. In this connection, we have studied the possibilities of using modifying additives, as a result of which it will receive identical bricks in terms of color characteristics. As is known, in order to obtain a dense, high-strength, and chemically stable ceramic brick, the ceramic mass is modified with an active component that promotes the formation of mullite. Such material includes active silica, which is an amorphous material of biogenic origin.

RESULTS AND DISCUSSIONS

The raw materials for the development of materials for the restoration of architectural monuments were loess-like rocks of the Suzanlinskoye deposit, sugar production waste-defecation formed at the JV JSC “Khorezm Shakar” and amorphous silica obtained by roasting rice husks.

The Suzanli field is located in the Khiva region of the Khorezm region. The quarry is located 4 km southeast of the village of Kasma on the territory adjacent to the Karakum desert, with a total land area of 28 hectares. The quarry of the deposit is located on a geologically flat surface, and the thickness of the reservoir is 3.0-4.5 meters. This deposit was formed on the site of the ancient Amudarya delta. In terms of the geological structure, the deposit corresponds to the alluvial deposits (Upper Quaternary layer) of the Akchadarya complex and consists mainly of sand, sandy loam, and loess-like rocks. The total reserves of the deposit are 829 thousand m³.

Loess-like rocks are mostly light brown or gray in color, consisting of minerals that form a complex structure. According to the data obtained, a comprehensive study of loess-like rocks is of great scientific and practical importance, since more than 70% of the territory of the Republic of Uzbekistan is loess and loess-like rocks.

Table 1. Results of chemical analysis of loess-like rocks of the Suzanli field

№	Chemical composition, wt. %									
	SiO ₂	Al ₂ O ₃	CaO	MgO	Fe ₂ O ₃	K ₂ O	Na ₂ O	SO ₃	TiO ₂	LOI
1	52,54	14,72	14,04	4,70	7,20	2,76	2,12	0,18	0,68	1,06
2	52,55	15,60	14,09	4,65	7,44	2,78	2,08	0,11	0,66	0,04
3	52,53	15,59	14,32	4,52	7,27	2,82	2,08	0,14	0,69	0,04
4	52,49	15,27	13,31	4,62	7,25	2,75	2,11	0,14	0,64	1,42
5	52,56	14,56	14,14	4,53	7,44	2,71	2,02	0,13	0,65	1,25
6	52,60	15,72	14,27	4,49	7,20	2,84	2,15	0,17	0,50	0,06
7	52,57	15,39	13,48	4,48	7,26	2,72	2,05	0,12	0,62	1,31
8	52,51	15,57	14,11	4,61	7,23	2,85	2,14	0,15	0,63	0,20
9	52,65	15,54	13,65	4,43	7,21	2,81	2,28	0,16	0,61	0,66
10	52,50	14,51	14,55	4,42	7,22	2,74	2,07	0,12	0,67	1,20
average	52,55	15,36	13,99	4,55	7,28	2,78	2,11	0,14	0,64	0,72

The study of clay raw materials of loess-like rocks was carried out in accordance with GOST 21216–2014. Scientific research to determine the chemical composition of clay from the Suzanlinskoye deposit was carried out in the laboratories of St. Petersburg State Technological University. According to the results, depending on the amount of Al_2O_3 in the raw material, the sample belongs to the group of acidic clay raw materials, and according to the amount of TiO_2 and Fe_2O_3 – to the group of coloring raw materials. In the production of ceramic building materials, the amount of SiO_2 in the raw material is limited to certain standards. The content of Al_2O_3 in the raw material used in the experimental work is 15.36% on average. This amount is not enough to get high-quality ceramic bricks. The content of CaO (13.99%), and MgO (0.17%) – indicates the presence of carbonate inclusions such as calcite and dolomite, which, in turn, reduce the sintering temperature during the firing of ceramic bricks [18].

The study of the granulometric composition of the loess-like rock of the Suzanlinskoye deposit was carried out on the Shimadzu SALD–7500 instrument. The granulometric composition of raw materials was determined on the basis of laser diffraction by dry and wet analysis. The results of the study are presented in Fig. 1.

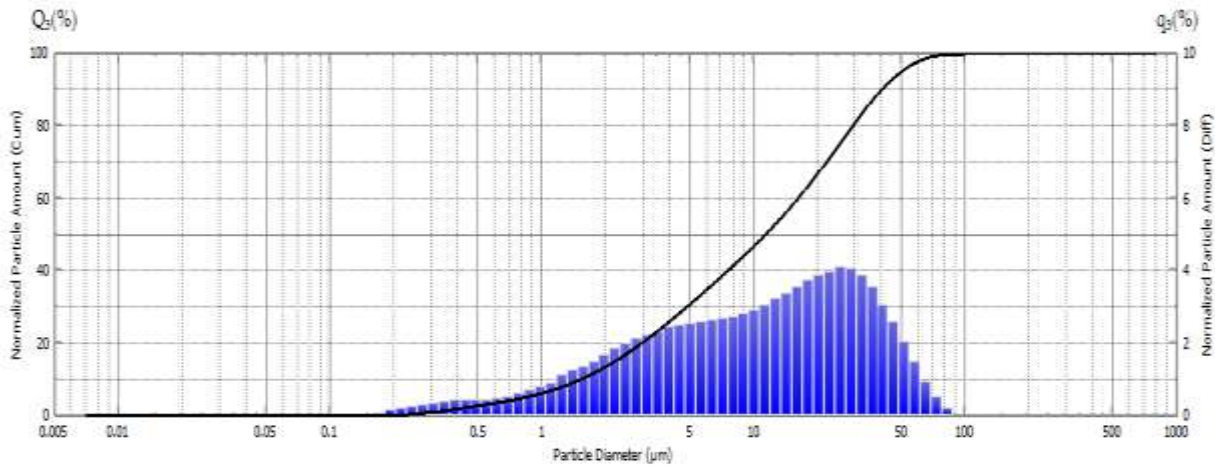


Fig.1. Granulometric composition of loess-like rocks of the Suzanli field.

According to the results of the experiment, it was established that the particle size distribution in the raw material is bimodal with maxima at about 3.5 μm and 25 μm . The average particle size is about 9 μm [7].

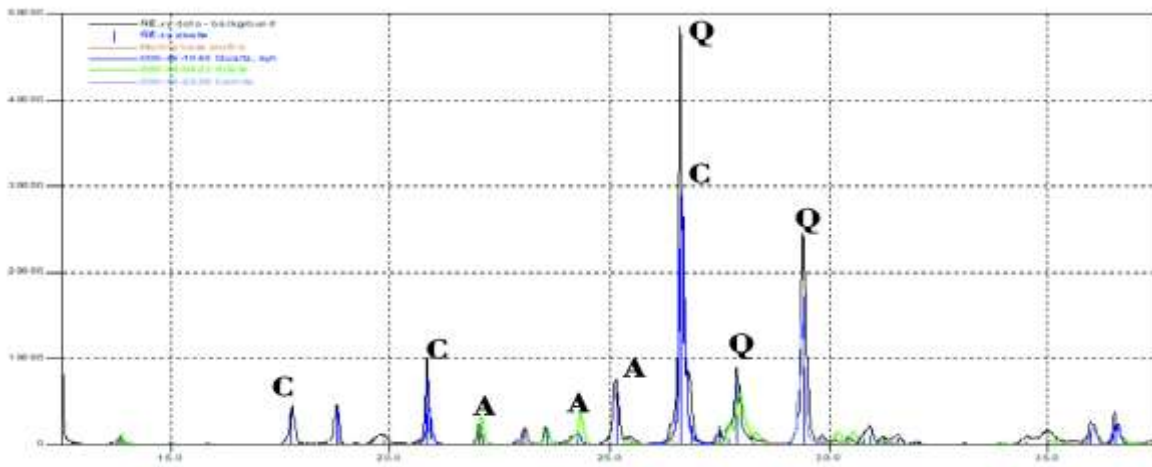


Fig.2. X-ray phase analysis of loess-like rocks of the Suzanli field

According to the results of radiographs of the main phases of the loess-like rocks of the Suzanlinskoye deposit (Fig. 3), the raw material contains quartz (SiO_2) ($d/n = 4.2592; 3.3448; 0.095; 2.4573; 2.2827; 2.2379; 1.9811; 1.8132; 1.6726; 1.6600; 1.5423;$

1.4536; 1.3826; 1.3755; 1.3722; 1.2563; 1.2001 Å), calcite (CaCO₃) (d/n =4.0344; 3.7771; 3.1940; 3.0360; 2.8214; 2.4573; 2.2827; 2.2379; 2.1288; 1.9954 Å), albite (Na (Al Si₃ O₈)) (d/n =3.0360; 2.2827; 2.0939; 1.1806; 1.1533 Å).

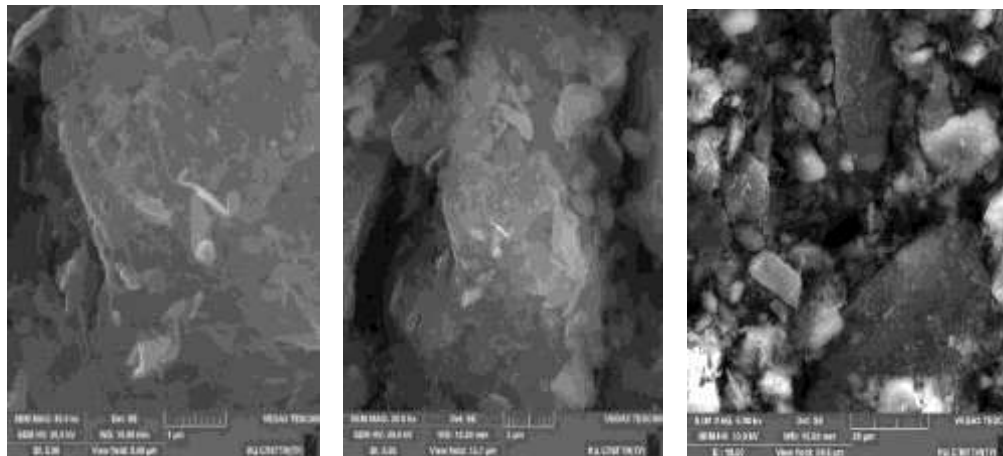


Fig.3. Image of electron microscopic analysis of loess-like rocks of the Suzanli field

The results of electron microscopic analysis of loess-like rocks, which are raw materials for the Suzanlinskoye deposit, are shown in Fig. 3. in an isometric view. Hexagonal shapes in the cloud refer to the mineral calcite, granules in the form of ovals, to the mineral quartz, and granules in the form of black spots, to the mineral albite.

The samples were also subjected to differential thermal analysis. The description of the obtained results is presented in Fig. 4. four.

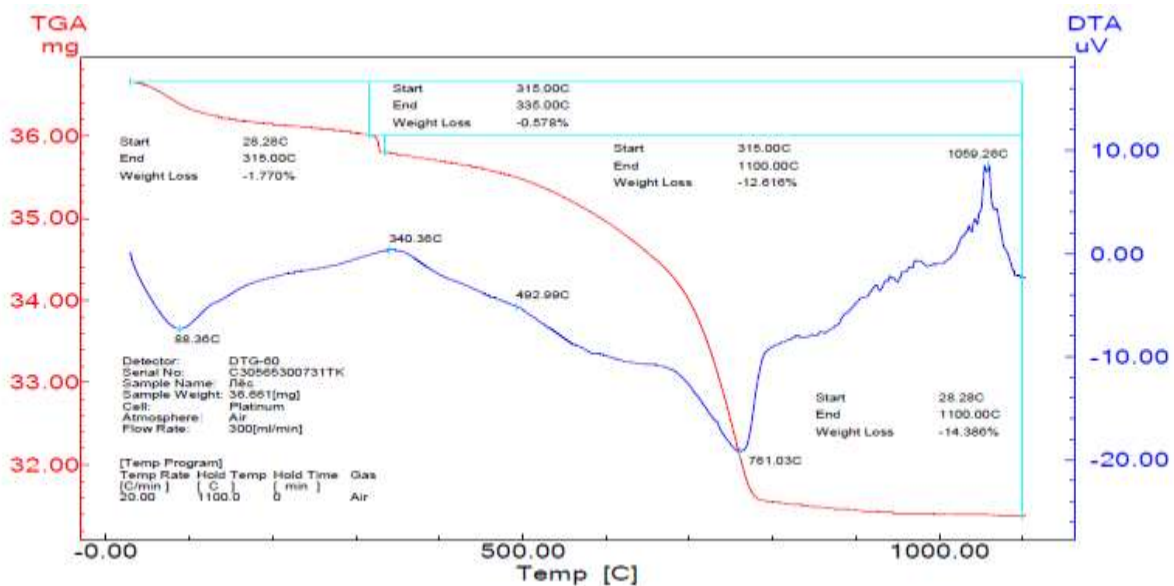


Fig.4. Derivatogram of less-like rocks of the Suzanli deposit

Differential thermal analysis of loess-like rocks showed the presence of an endothermic effect at temperatures of 88°C and weight loss corresponds to the evaporation of water. Exothermic effects at a temperature of 340°C – refer to the burnout of organic impurities. The endothermic effect at a temperature of 761°C and the main mass loss corresponds to the decomposition of clay minerals and carbonates. The exothermic effect at a temperature of 1059°C – synthesis or crystallization of new compounds of calcium silicates.



The technological properties of the samples are as follows:

- fire resistance – 1150 °C;
- plasticity according to Atterberg – 9–10;
- coefficient of propensity to dry out – 180 s;
- mechanical strength of dried samples – 2.5–2.6 MPa;
- average density – 1470–1480 kg/m³.

Other disadvantages of ceramic bricks made of loess-like silica include their low frost resistance. As indicated in [20] by studies conducted over a number of years in the laboratory of YuzhNIStrom, it was found that in order to achieve high frost resistance of ceramic stone of semi-dry pressing (without worsening its appearance) and the possibility of obtaining a solid brick, it is necessary to introduce a mineral additive that helps optimize the structure of the stone in the roasting process. The authors used calcium-containing waste generated during the production of mineral fertilizers as an additive to increase the frost resistance of ceramic stone. The results obtained with the introduction of mineral additives allowed the authors not only to increase the frost resistance index of products above those required by GOST 530–2012 but also to improve their appearance and strength. Other sources report that to increase the frost resistance of ceramic bricks [21–23], modifying additives. The authors attributed calcium-containing components to such additives. In another work, the authors indicate that on the basis of clay raw materials from the Kagalnik–3 deposit with the addition of 1% by weight of coal of the ASH grade, according to the semi-dry pressing scheme, it is possible to obtain ceramic bricks with a strength grade of M100–125 and frost resistance of 15 cycles [24].

Based on this, we also chose a mineral additive to obtain high-quality restoration bricks with increased frost resistance from loess-like loam, which was taken as defecation waste from sugar production. Defecation is a cream-colored powder, greasy to the touch, easily moistened with water, forming a plastic dough. Defecation humidity after extraction from filters is 20.00–24.00%, density 2135–2380 kg/m³, specific volume 1030–1095 kg/m³. The results of the study of the material composition of the defecation of JSC “Khorazm Shakar” show that the following ingredients are present in its composition (in wt.%): sugar – 1.60–2.10; pectin substances – 1.40–1.65; calcium carbonate – 74.25–80.00; nitrogenous organic substances – 5.90–6.01; without nitrogenous organic substances – 8.04–9.50; lime in the form of salts of various acids – 2.80–3.10; other mineral substances – 2.78–3.91. Table 2 shows the results of studying the chemical composition of the defecation.

Table 2. Chemical composition of the defecation of JSC “Khorazm Shakar”

samples	The content of oxides, wt. %									
	CaO	MgO	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	SO ₃	R ₂ O	p.l	other	Sum
D–1	48,43	1,17	0,43	2,65	0,58	0,53	0,05	45,70	0,56	100
D–2	48,41	1,15	0,41	2,67	0,55	0,51	0,07	45,60	0,63	100

As can be seen from table 2, the composition of the defecation is identical to the calcium-containing natural compound. The existing organic matter contributes to the organization of the reduction mode of firing, as a result of which it is possible to reduce the firing temperature by 50°C, this is described in detail in the work of the authors [20].

The study of the particle size distribution of the waste of sugar production – defecation on the Shimadzu SALD–7500 device (Wing SALD II: Version 3.4.1). The granulometric composition of the defecation was determined on the basis of laser diffraction by the method of dry and wet analysis. The results of the study are presented in Fig.5.

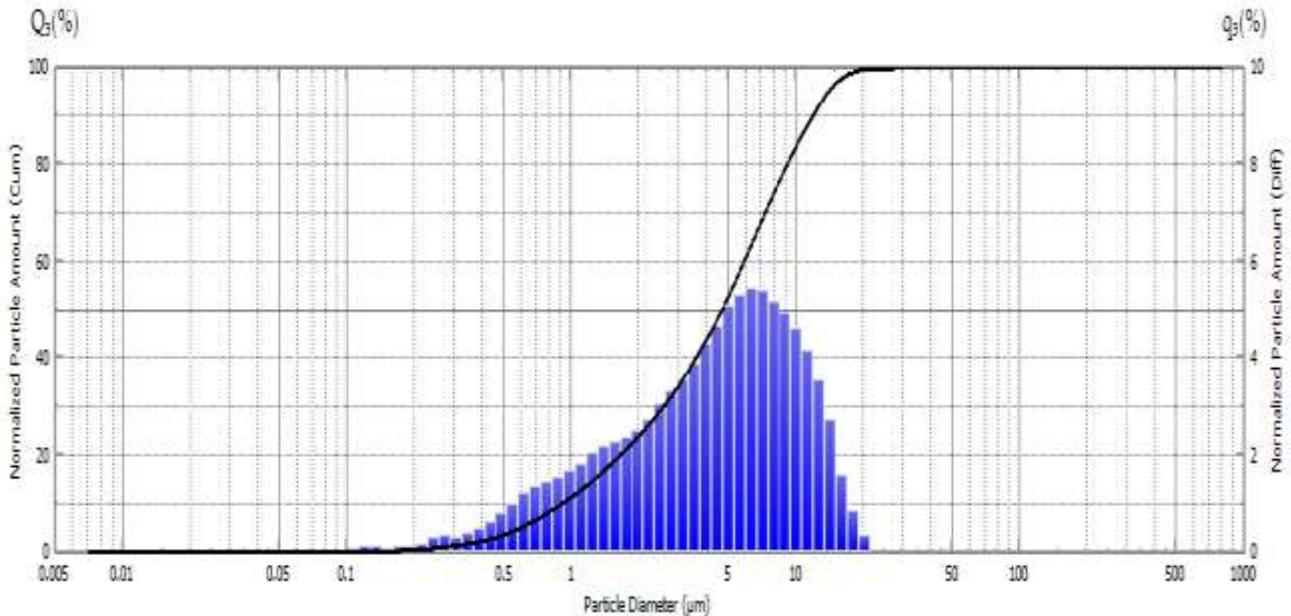


Fig.5. Granulometric composition of defecation

The size distribution of particles is practically unimodal (one effect) with a broadening towards fine particles. The average particle size is about 3.8 microns. Modal (the maximum of the greatest effect is about 6.8 microns. The maximum particle size does not exceed 30 microns.

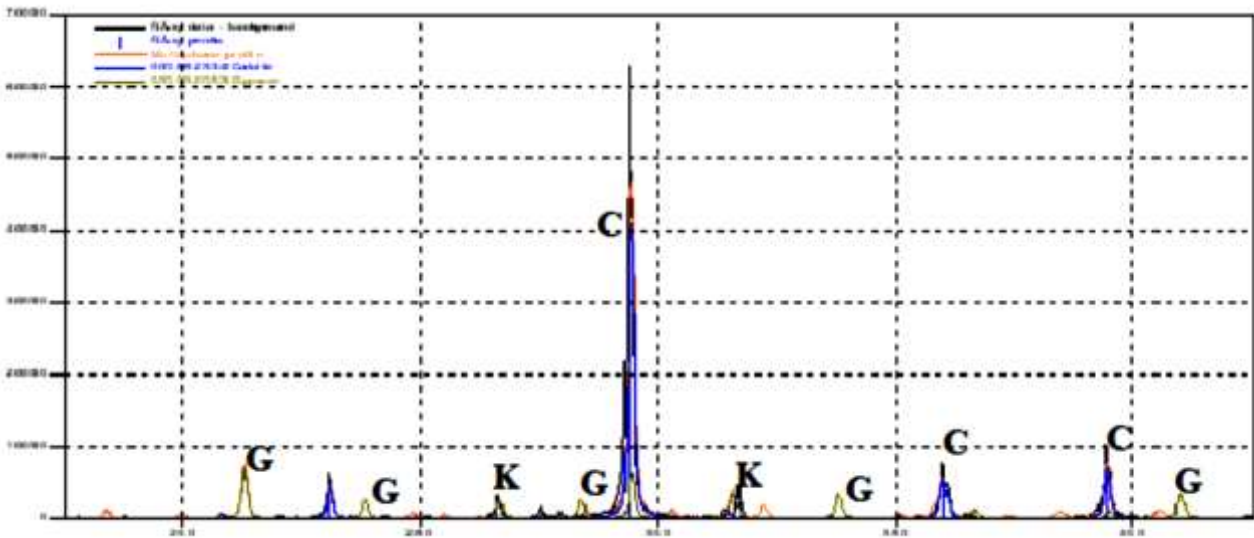


Fig.6. X-ray phase analysis of sugar defecation

According to the results of radiographs of the main phases of the defecation (Fig. 6), the raw material contains calcite ($-d/n = 4.2583; 3.3455; 2.4582; 2.2376; 2.1285; 1.9805; 1.8184; 1.6722; 1.5419; 1.3823; 1.3752; 1.3721; 1.2563; 1.2000 \text{ \AA}$), kaolinite ($-d/n = 6.3873; 3.7765; 3.3712; 3.1927; 2.4942; 2.4582; 1.9805; 1.8184; 1.6722 \text{ \AA}$), gypsum ($d/n = 3.0344; 2.4942; 2.0939 \text{ \AA}$).

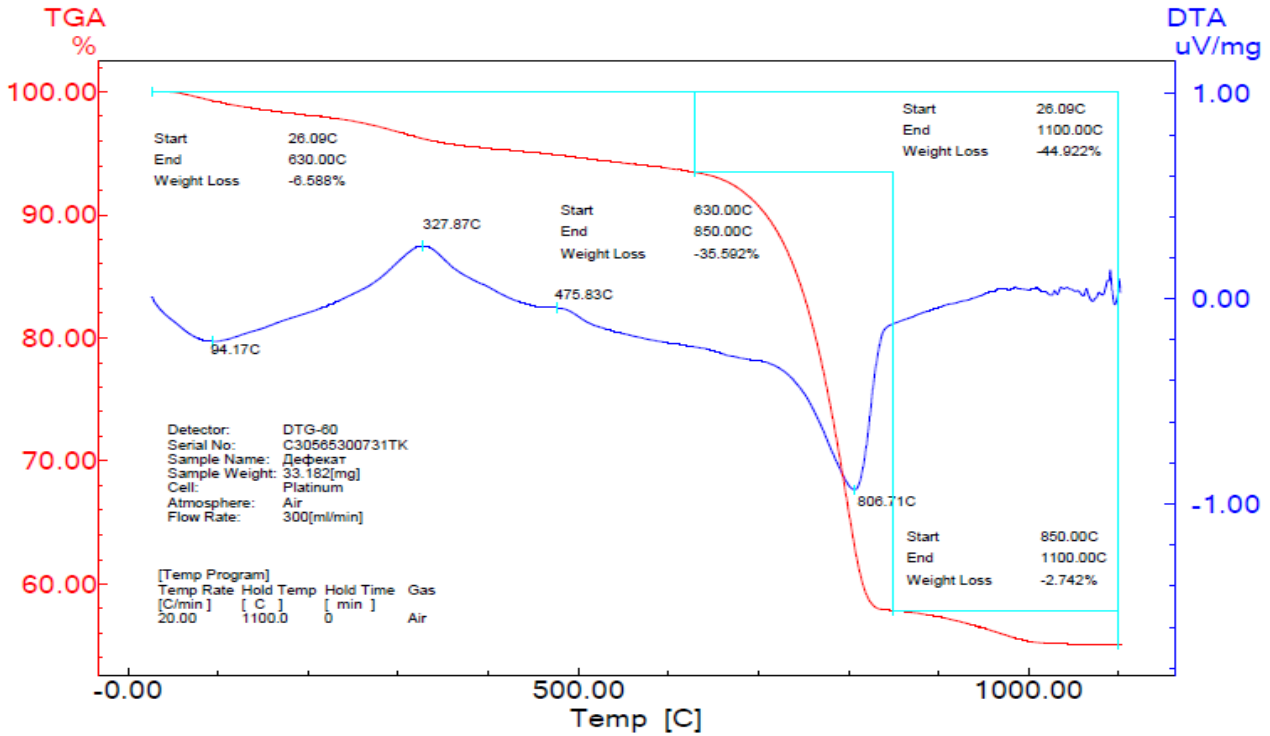


Fig.7. Derivatogram of the defecation

The differential thermal analysis of the defecation shows an endothermic effect at 94°C and weight loss – water evaporation. Exothermic effects at 327°C and 475°C – burnout of organic matter. The endothermic effect at 806°C and the main mass loss associated with it is the decomposition of calcium carbonate. Weight loss after 850°C – decomposition of impurities in the defecation (Fig.7).

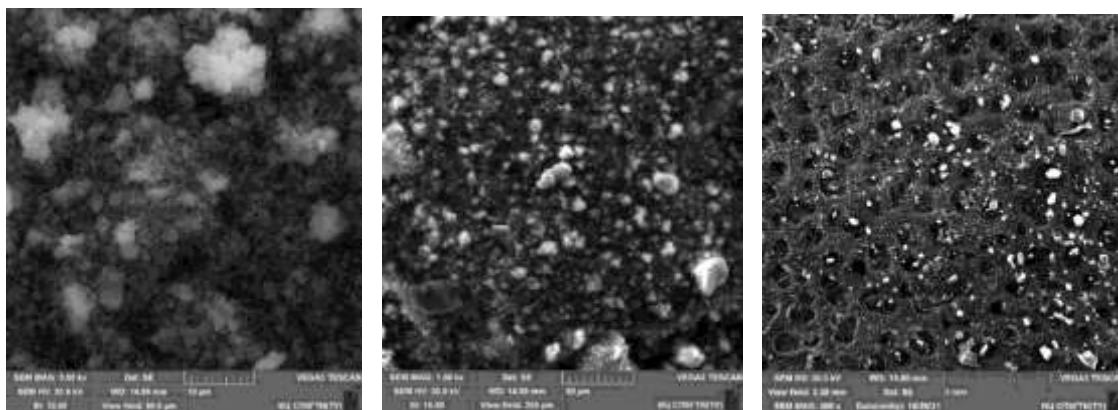


Fig.8. Image of electron microscopic analysis of defecation

The results of the study on the electron microscopic analysis of the defecation are shown in fig. 8., reflected in the isometric image, hexagonal-shaped granules with broken cloud sides belong to the calcite mineral, oval-shaped granules to the gypsum mineral, and granules in the form of black spots to kaolinite.

**Table 3.** Chemical composition of amorphous silicon waste, %

Sample	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	RO	R ₂ O	SO ₃	Cr ₂ O ₃
Amorphous silica	71,0	3,5	0,6	10,5	14,0	0,3	0,1
	70,6	5,0	0,8	9,1	14,1	0,4	–

Thus, the set of main impurity elements present in the studied silica samples, as in [25, 26], includes calcium, magnesium, aluminum, iron, and manganese. The content of their oxides depends on the purity of silica (tables 6–8) and varies within the following limits (%): iron oxide 0.090–0.021, aluminum 0.064–0.11, calcium 0.12–1.1, magnesium 0.023–0.058, manganese 0.0001–0.340. The smallest amount of the sum of impurity oxides was found in samples of amorphous silica obtained according to schemes 2 and 3. According to [25], in addition to the oxides of iron, aluminum, calcium, magnesium, and manganese, oxides of the following elements are present in the samples of silicon dioxide: Na, K, Rb, Cu, Ag, Zn, and P; however, their amount is insignificant, as in [25]; therefore, in this work, their effect on the properties of the obtained samples of amorphous silica was not studied.

CONCLUSIONS

In conclusion of these studies, it should be noted an important circumstance that this waste of sugar production – defecation and amorphous silica in its natural form contribute to the formation of anorthite in a ceramic shard, as a result of which mechanical and chemical properties improve, and the organic part of the defecation contributes to the formation of a reducing firing mode, as a result, which turned out to be a shard that corresponds to the color characteristics of old bricks.

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