Journal of
Composites Science

MDPI

Article

Strong Structure Formation of Ceramic Composites Based on Coal Mining Overburden Rocks

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Abstract: Currently, the amount of man-made waste worldwide is steadily increasing. It is, therefore, necessary to constantly look for effective ways of utilization and recycling. It is also necessary to reduce the use of non-renewable resources and reduce the impact on the environment. The use of coal industry waste is currently quite insignificant, amounting to some 10% of the total volume. The work aimed to study the properties of raw materials and study the processes of structure formation during the forming, drying, and firing of composite ceramic produced using overburden rock and additives. The work's relevance lies in the need to solve environmental, economic, and technological problems related to the utilization of coal mining waste. Experiments of the past prove the possibility of using the waste coal industry as additives in the production of building materials. The article presents the results of studies of the chemical, mineralogical, and granulometric composition of overburden rock in coal mining. Peculiarities of structure formation during the forming, drying, and firing of ceramic composites based on optimal fractional compositions from coal-mine overburden were revealed. Organic and chemical additives were used for the correction of technological properties and improvement of the quality of finished composite products. The physical and mechanical indices of the obtained composite ceramic samples were determined, the analysis of which revealed that the use of highly mineralized carbonaceous rocks as solid additives provided a 2–2.5-fold increase in the strength of the product, 5.6% reduction in water absorption, and an increase in the product frost resistance by 20–25 cycles. The aluminum oxychloride influence on the physical and mechanical indices of the obtained composite articles was reflected in a reduction in their water absorption from 8.2 to 7.0%, a 10–12% increase in strength in compression, and an increase in freeze–thaw resistance by 30–35 cycles. Research results proved that the composition and properties of coal-mine overburden rock are close to those of conventional clays. With special technological preparation, they can be used for the production of composite ceramic products. This will significantly reduce the cost of bricks, to make up for the shortage of high-grade clay raw materials and improve the environmental situation. Nevertheless, further research into the use of coal-mine overburdens in the composite ceramic material technology is warranted.

Keywords: overburden rock; composition; properties; molding; drying; firing; ceramic composites

Citation: Rakhimova, G.; Stolboushkin, A.; Vyshar, O.; Stanevich, V.; Rakhimov, M.; Kozlov, P. Strong Structure Formation of Ceramic Composites Based on Coal Mining Overburden Rocks. *J. Compos. Sci.* **2023**, *7*, 209. https://doi.org/10.3390/jcs7050209

Academic Editor: Jacques Lamon

Received: 8 April 2023
Revised: 29 April 2023
Accepted: 18 May 2023
Published: 22 May 2023

1. Introduction

With the growth of manufacturing, the amount of industrial waste in the world is constantly increasing [1,2]. Only 13.5% of waste is recycled worldwide, and more than 33% of waste is improperly disposed of, according to a World Bank report [3]. Therefore, it is necessary to constantly look for effective ways of utilization and recycling. It is also important to reduce the use of non-renewable resources and reduce the impact on the environment. One of the ways to dispose of solid waste of the fuel and energy complex is to use it as construction materials or raw materials for their production [4]. Studies carried out in the United States, Western Europe, Japan, and other countries attest to the possibility and expediency of the production of various ceramic products with the use of technogenic wastes, such as ash, slag, coal-mine overburden, and others [5]. Studies of the past few years demonstrated the possibility of using coal industry wastes as burning and baking additives in the production of ceramic bricks [6,7].

In the process of open-pit coal mining at the world's largest deposit in Ekibastuz (Kazakhstan), a huge amount of waste—overburden—is generated and disposed to waste dumps. Currently, approximately 4 billion cubic meters of overburden are being accumulated at dump sites. Coal-mine overburden dumps reach heights of up to 100 m and occupy large areas of land near the city. Coal mine waste worsens the environmental situation in the region and is a source of dust storms, fires, and gas pollution.

Overburden rocks of coal mining are represented mainly by argillites and siltstones. They differ from other types of mineral raw materials in the content of organic matter. Waste coal mines have peculiarities of physical–mechanical and physical–chemical properties as a result of the carbonization and metamorphism of rocks [8–10]. The widespread use of coal waste is hampered by insufficient knowledge of its properties. There are no scientifically substantiated technologies of coal waste processing or methods for evaluating indicators of their technological properties. [11,12]. Due to the material composition, overburden rocks of coal mines are promising raw materials for the production of ceramic building materials. Currently, coal mine waste is used in brick factories in the form of additives in the production of wall ceramics [13].

It is known that creating composite products for construction ceramics involves the formation of three types of structures: coagulation structures, condensation-crystallization structures, and crystallization structures [14]. A coagulation structure is formed when preparing ceramic masses and forming products. The transition from coagulation structure to condensation-crystallization structure occurs when water is removed during the drying of products made using plastic molding. In the process of heat treatment (firing), the formation of the crystallization structure of ceramic products takes place [15,16]. Experimental studies into different types of structures formed in the production of composite products were conducted to a greater extent for clay [17–20].

The processes of formation of coagulation, condensation, and crystallization structure during forming, drying, and firing of ceramics using coal-mine overburden are poorly studied, and so, research in this area is quite relevant. The aim of the work is a comprehensive study of the processes of structure formation during molding, drying, and firing of wall ceramic composites made from coal-mine overburden to obtain defect-free ceramic products. Research objectives: study chemical, granulometric, and mineral composition of coal-mine overburden; define and control the degree of influence of the fractional composition of masses on properties of samples during molding; study condensation structure formation during the drying of ceramic bricks made from coal mining waste; study the formation of crystal structure in the process of firing ceramic bricks from coal mining waste; study burnout kinetics of organic matter from coal mining waste in the process of its firing.

The novelty and significance of the research includes a comprehensive study of overburden rocks of coal mining as the main, environmentally safe raw materials for obtaining composite ceramic products being conducted. Features of structure formation at forming, drying, and firing of ceramic composites based on optimum fractional compositions are revealed. Organic and chemical additives are used for the correction of technological properties and improvement of the quality of finished products. Resource-saving compositions and energy-efficient production technology of composite building ceramics are developed. The studies are of practical value for the enterprises of the coal industry producing overburdened rocks in the form of production wastes. They can significantly reduce the cost of transportation, and storage of waste, reduce environmental payments, reduce the cost of production and make up for the lack of high-grade clay raw materials in the production of ceramic products, and improve the environmental situation in the region.

2. Materials and Methods

2.1. Materials

When stripped of the useful layer of coal deposits using the open-pit method, overburden is formed, which is represented by sedimentary rocks—clays, argillites, loams, siltstones, conglomerates, sands, gravels, and shales. Studies of overburden composition showed that argillites and siltstones were the prevailing rocks in coal mining waste. Clay minerals in argillite and siltstones were mainly represented by hydromica, kaolinite, and montmorillonite. A distinctive feature of overburdens is the presence in them of residual coal, residing in intergrowths with mineral components.

Figure 1 shows the estimated amount of man-made waste produced by enterprises in the Pavlodar region (Kazakhstan) for 2019 [21]. The volume of technogenic waste allows us to talk about the possibility of the emergence of a serious raw material base for the production of building products.

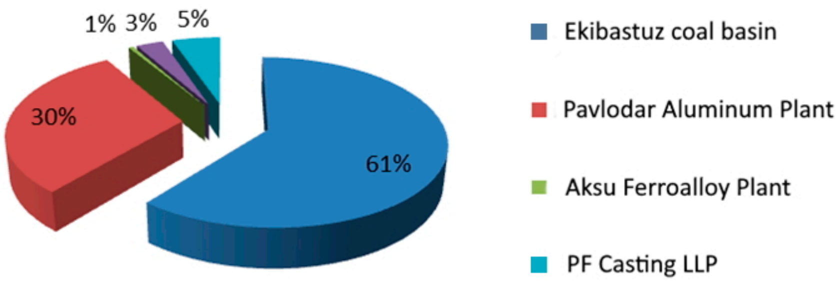


Figure 1. Estimated amount of man-made waste (2019).

Overburden of the Ekibastuz coal basin comprises argillite, siltstone, and coal shale, and constitutes a valuable organometal material. In terms of properties, argillites and siltstones are similar to traditional clays and can be used as raw materials for the production of ceramic building materials [22]. The use of argillites and siltstones will reduce energy and production costs, and improve the technical characteristics of the products.

Compared with traditional clay raw materials, argillites and siltstones have their specific features, which must be taken into account when evaluating them as raw materials for the production of ceramic products (Figure 2). They have a high average density (2.55–2.70 g/cm^3), which largely depends on the presence of cracks. True density is 2.69–2.74 g/cm^3. Porosity is 1–4%, water absorption is on average 2–5%. When dry, argillites and siltstones (Figure 2) exhibit compressive strength (5–20 MPa). When moistened, the strength decreases sharply. The color of most types of argillites is gray or dark gray. The structure is aleuropelitic. The texture is oriented, layered, and, in some places, disorderly. Under a microscope, transmitted light showed mica flakes oriented along layering planes.

Overburden rocks from coal projects have certain physical and mechanical properties which depend on the degree of metamorphism of rocks. In their natural form, coal mine wastes are not soaked in water, which requires mechanical grinding to break the cementation bonds of the clay components.

In general, characteristics of argillites and siltstones in terms of structural and physical composition and mechanical properties are comparable to other types of clay raw materials. This suggests their potential suitability as a raw material for building ceramics. It is necessary to develop an appropriate methodology for their evaluation, testing, and production technology.

(**a**) (**b**) (**c**)

Figure 2. Overburden in the Ekibastuz coal basin: (**a**)—argillite; (**b**)—siltstone; (**c**)—coal shale.

2.2. Research Methods

Standard methodology for physical and mechanical tests and precision research methods: X-ray powder diffractometry, infrared absorption spectroscopy, scanning electron microscopy, quartz dilatometry, etc., were used in the work [23,24].

The studies were carried out at the Karaganda Technical University in Karaganda (Kazakhstan) and the Siberian State Industrial University in Novokuznetsk (Russia). The study of the particle size distribution was carried out by a sieve method of analysis, as well as by a laser particle size analyzer Malvern Mastersizer 2000 (Malvern, UK). Particle measurements were carried out in wet and dry types in the range from 0.02 to 2000 microns. The chemical composition of the starting materials was determined by qualitative spectral analysis on a Shimadzu XRF-1800 X-ray fluorescence wave dispersive spectrometer (Tokyo, Japan). For research of parameters of structure and phase composition of raw components and fired ceramic products, the complex of precision and direct methods, including qualitative X-ray diffraction analysis on diffractometer DRON-3. 0 (Moscow, Russia), X-ray diffraction analysis on X-ray diffractometer Shimadzu XRD-6000 (Tokyo, Japan), differential thermal analysis and thermogravimetry on simultaneous thermal analysis unit Setaram LabSys Evo (Caluire, France), infrared spectroscopy on spectrophotometer Shimadzu IRAfinity-1 (Tokyo, Japan), optical and electron microscopy using Olympus GX-51 optical microscope with polarization attachment (Tokyo, Japan) and JSM-6460LV scanning electron microscope (Tokyo, Japan) with the system of energy dispersive microanalysis Oxford INCA Energy (Hertfordshire, United Kingdom) were used.

According to the data from the chemical analysis of overburden of different lithological types, we concluded on the quantitative content of rock-forming oxides (Table 1). According to free quartz content, coal mining waste belongs to the group of raw materials with average quartz content.

According to its content of aluminum oxide $A1_2O_3$ in the calcined state, all the studied rocks belonged to the group of semi-acidic raw materials.

Iron compounds are represented mainly by pyrite and siderite. By the content of iron oxides, overburden rocks belong to the group of raw materials with high content of dyeing oxides.

The total content of calcium and magnesium oxides was 1.67–2.3% for argillites and 1.09–1.84% for siltstones.

The alkaline oxides of sodium and potassium were mainly present in clay-forming minerals and were partially present in admixtures in the form of water-soluble salts.

In terms of sulfur oxide SO_3 content, overburden rocks belonged to the group of low-sulfur, environmentally safe raw materials, which allowed for their use in the production of ceramic products without restrictions. Additionally, overburden rocks contain organic carbon.

Table 1. Chemical composition of the samples studied.

Raw Material	Mass Content, % (After Ignition)											
	SiO_2	TiO_2	Al_2O_3	$Fe_2O_3 + FeO$	MgO	MnO	CaO	Na_2O	K_2O	P_2O_5	SO_3	Ignition Loss
Horizon +50 m												
Argillite	56.7	0.9	17.6	5.6	1.1	0.1	0.2	1.2	1.9	0.25	0.15	12.05
Siltstone	61.1	1.03	18.5	3.4	0.9	0.12	0.85	1.32	2.06	0.25	0.21	10.4
Horizon +100 m												
Argillite	57.7	0.85	17.7	6.4	1.2	0.1	0.77	0.57	2.39	0.3	0.10	11.0
Siltstone	59.9	0.93	17.96	4.18	1.1	0.09	0.74	1.06	2.24	0.25	0.22	10.9
Horizon +150 m												
Argillite	57.7	0.86	18.4	5.6	0.94	0.12	0.73	0.47	2.54	0.38	0.19	11.6
Siltstone	61.3	0.93	17.6	4.02	0.03	0.08	0.56	1.38	2.11	0.28	0.28	10.06

The mineral composition of the overburden rocks in question in relation to the lithology and horizon of occurrence is presented in Table 2.

Table 2. Mineral composition of overburden rocks.

Name of Raw Material	Mineral Content, %							
	Clay Minerals		Quartz	Feldspar	Carbonates	Mica	Siderite	Organic Material
	Kaolinite	Hydromica						
Horizon +50 m								
Argillite	25–30	20	25	8–12	-	-	-	15
Siltstone	5–10	25	30	20–25	3–5	5	-	10
Horizon +100 m								
Argillite	25	20	30	-	-	-	5	12
Siltstone	5–7	20	40	25	3–5	5	-	8
Horizon +150 m								
Argillite	15	20	40	10	-	-	-	10
Siltstone	3–5	20	50	15–20	5	-	-	5

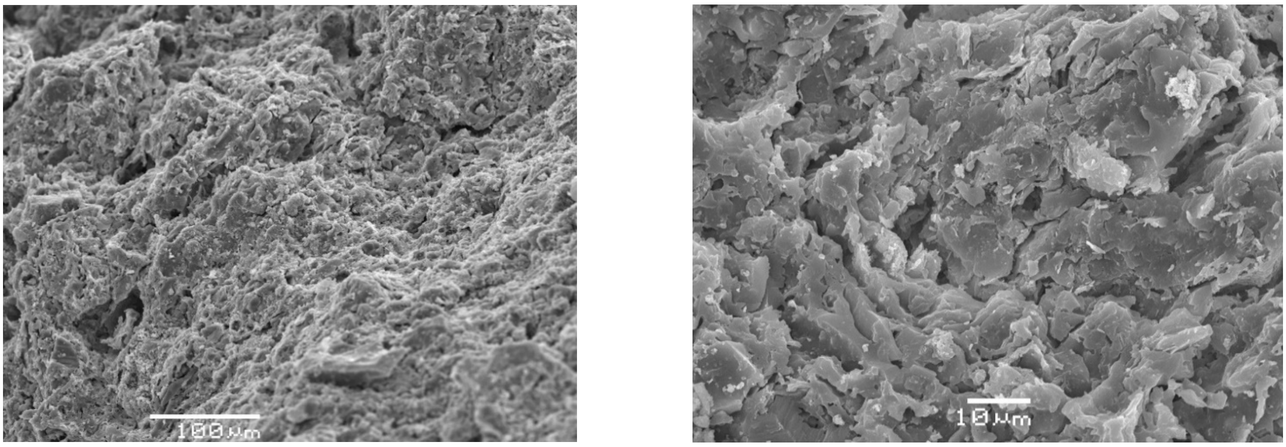
According to the mineralogy of the clay component, overburden rocks belong to kaolinite-hydrosludite type raw materials.

Among the nonclay minerals, the examined samples contained impurities of quartz, feldspar, mixed-layer minerals, as well as organic substances. They can be used as a basic raw material for the production of ceramic building materials [25,26].

Distinctive areas of the microstructure of overburden rocks were studied using a scanning electron microscope (Figure 3). The microtexture of the argillite was uniform and had individual larger aggregates of irregular shape, mainly 20–50 microns in size.

In addition to X-ray diffractometry by international crystallographic and crystal-chemical databases on minerals and their structural analogs, infrared absorption spectra of overburden argillites and siltstones were studied (Figure 4). In the low- and medium-frequency regions of the spectrum (up to 1800 cm^{-1}), the composite material had absorption maxima of 470, 545, 1090, and 1170 cm^{-1}, which is characteristic of hematite. The presence of quartz was confirmed by the characteristic doublet 770, 790 cm^{-1}. Absorption maxima (605, 1090 cm^{-1}) corresponded to mullite.

Overburden rocks by their particle size distribution were medium-dispersed raw materials, as the medium-dispersed grains in argillites was 45.5–53.2%, and in siltstones 27.0–35.2% (Table 3). Analyzing the granulometric composition of overburden rocks on the basis of "lithological type", it should be noted that the content of clay particles in argillites was 2–8% higher than in siltstones, and sandy fraction prevailed in siltstones, with its content being 19–26% higher than in argillites.



(**a**) (**b**)

Figure 3. Micrographs of the structure of the argillite of the +100 m horizon, magnification, respectively: 150× (**a**); 750× (**b**), scanning electron microscope.

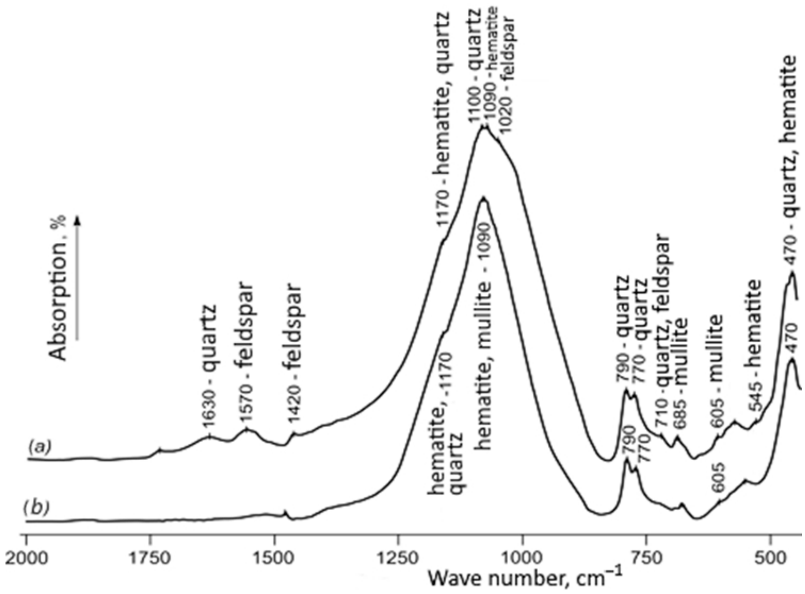


Figure 4. Infrared absorption spectra of argillite (**a**) and siltstone (**b**) burnt at 1000 °C.

Table 3. Granulometric composition of overburden rocks.

Material	**Quantitative Content of Particles, %**			**Variety of Raw Materials According to Okhotin's Diagram**
	Finely Dispersed 5 μm	**Medium-Dispersed 5–50 Microns**	**Coarsely-Dispersed 50–1000 Microns**	
		Horizon +50 m		
Argillite	29.8	53.2	17.0	Silty clay
Siltstone	21.6	35.2	43.3	Silty loam
		Horizon +100 m		
Argillite	20.4	49.6	30.0	Silty clay
Siltstone	18.1	31.6	50.3	Silty loam
		Horizon +150 m		
Argillite	14.3	45.5	41.2	Silty clay
Siltstone	12.5	27.0	60.5	Silty loam

The predominance of medium- and coarsely-dispersed particles in argillites allowed them to be classified as dusty clays, and siltstones as dusty loams, since the quantitative content of particles ranging from 5 to 1000 microns in them was within 78.5–87.5% (Figure 5).

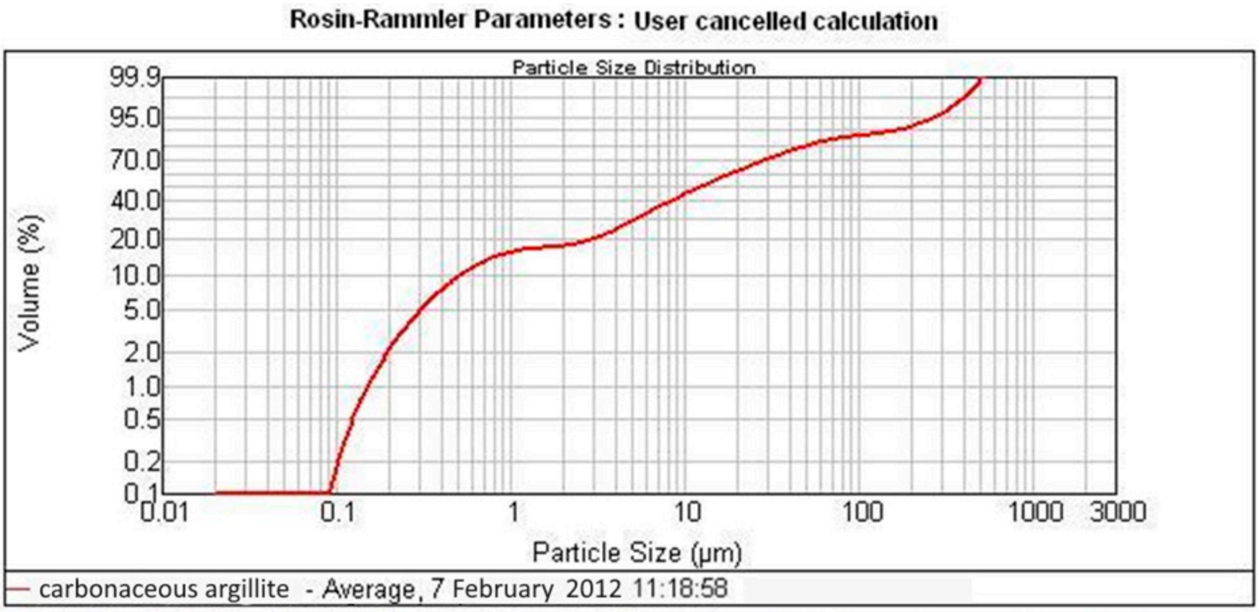


Figure 5. Dispersive composition of argillites.

The content of fine and medium-dispersed particles decreased for argillites and siltstones as horizons changed from +50 to +150 m, and the content of coarsely-dispersed particles increased, which confirmed the results of diffractometric and petrographic analyses showing a reduction in clay minerals and an increase in terrigenous materials—quartz, siderite, and feldspars—with the above change of overburden horizons from the roof of the coal seam.

3. Results and Discussion

3.1. Structure Shaping in the Ceramic Mass Forming Process

One of the most important technological operations in the production of ceramic bricks is mass preparation and shaping. At the forming stage, a coagulation structure is formed, which has certain technological and structural-mechanical properties, on which the quality of the finished products ultimately depends [27,28].

One of the most characteristic properties of clay materials is plasticity. Plastic properties of clay ceramic masses are evaluated using the Schwedow–Bingham equation [29]:

$$P = P_K + \eta_x * \omega \tag{1}$$

where P is applied shear stress causing steady flow of clay, Pa;

1. P_K is shear stress limit, Pa;
2. $\eta_{x;}$ is plastic viscosity;
3. ω is velocity gradient or relative shear rate.

It was practically established that well-formed clays are characterized by the following indicators of structural and mechanical properties: elasticity λ = 0.6–0.65; plasticity φ = 2.0-2.5-10-6 s^{-1}; true relaxation period θ = 1200–1400 s [30].

Thus, the specified structural–mechanical characteristics can serve as criteria for assessing the quality of ceramic masses. Using these characteristics, it is possible to control the structure and properties of masses in the process of their processing by introducing additives, the use of various technological methods that will ensure the production of products without defects in the structure with given optimum properties.

To determine the characteristics of elastic–viscoplastic properties of the masses of optimal molding moisture from overburden milled to particle size smaller than 0.5 mm, we used a device with parallel-sliding plates such as 6.MX.5C, the signal from which is transmitted through an amplifier to the potentiometer KSP-4 and samples (plates) are recorded on the media (Table 4).

Table 4. Structural and mechanical characteristics of overburden masses.

Feature	Designation, Units of Measurement	Horizon +50 m		Horizon +100 m		Horizon +150 m	
		Argillite	Siltstone	Argillite	Siltstone	Argillite	Siltstone
1	2	3	4	5	6	7	8
Humidity	Molding water content, %	17.5	17.1	17.5	17.0	16.6	16.3
Rapid elastic modulus of deformation	E_0, MPa	23.4	12.62	12.5	10.04	12.5	11.41
Modulus of slow elastic deformation	E_2, MPa	26.35	14.38	18.51	16.43	10.3	18.26
Conditional static yield strength	$P_{K1}10^{-3}$	0.1	0.14	0.21	0.2	0.24	0.23
Strain rate gradient	$\frac{dE^1}{d\tau}10^{-4}$	0.39	0.85	1.02	1.18	0.93	0.88
Plastic viscosity	$\eta_1 10^8$ Pa, s	534.2	393.3	470.4	375.6	436.3	351.4
Elasticity	λ	0.48	0.38	0.42	0.34	0.45	0.34
Plasticity per Volarovich	$\frac{P_{k1}}{\eta_1}10^{-7}$, s^{-1}	0.0022	0.0035	0.0044	0.053	0.057	0.065
The period of true relaxation	Θ, s	392	578	766	611	772	466
Rapid elastic deformation (elastic)	E_0, %	38	44.8	39.5	45.2	38	47.1
Slow elastic deformation	E_2, %	25.2	24.8	26.0	25.0	27.4	27.6
Plastic deformation	$E_1\tau$, %	36.8	30.4	34.4	29.8	34.6	25.3
Structurally mechanical type		III	III	III	III	III	III

Plastic masses must deform without disturbing the continuity and homogeneity and retain their shape after the load is removed. The limiting strength of the structure, at which there is a failure of its cohesion, is the plastic strength. To control the structural–mechanical properties of masses from overburden rocks and improve their coagulation structures, studies were conducted to optimize the fractional composition of masses by the simplex-scheme method of experiment planning, which made it possible, through a small number of experiments, to obtain a more complete characterization of the studied dependencies and determine the optimal ratio of particles. Experiments were conducted for three-component compositions, in which X_1, X_2, and X_3 were taken as the content of particles (%) of size 0.5–0.25; 0.25–0.125; 0.125–0.063 mm, respectively. The mathematical model of the dependence of structural–mechanical properties on the fractional composition of rocks was expressed by a polynomial of the second order:

$$y = b_1x_1 + b_2x_2 + b_3x_3 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 \quad (2)$$

1. y—measured parameter;
2. x_i—experimental values;
3. b_i—empirical coefficients.

Using a cone-and-plate rheometer, at a constant speed of cone immersion at 3 min, and with increasing loads following the scheme: 20, 70, 120, 170, 220, 270, 320 g, we studied the relationship between the plastic strength and moisture of the masses of overburden rocks of different fractional compositions. The research aimed to clarify the nature of the relationship between the value of plastic strength and moisture content of masses of argillite and siltstone horizons +50, +100, and +150 m, crushed to different grain sizes (Figure 6).

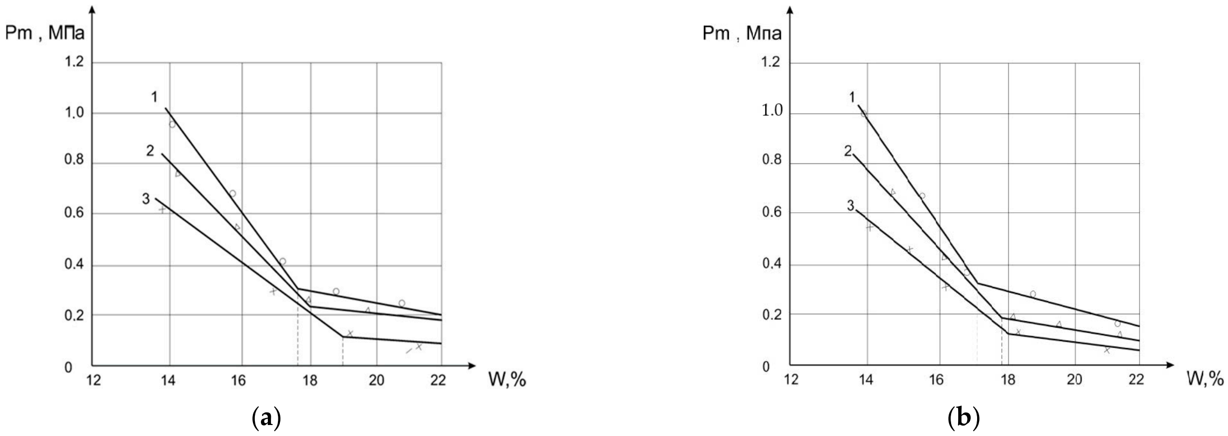


Figure 6. The curves of dependence of plastic strength on humidity of the masses: (**a**)—argillite horizon +100 m; (**b**)—siltstone horizon +100 m; 1-particle 0.5–0.25 mm; 2-particle 0.25–0.125 mm; 3-particle 0.125–0.063 mm.

Thus, the masses of rocks milled to a grain size of 0.5–0.25 mm were characterized by low development of plastic properties due to insufficient hydration, high strength, high energy consumption for processing, and can be classified as satisfactorily moldable. Masses of rocks 0.25–0.125 mm in grain size were well formed, sufficiently homogenized, and insensitive to deformations during drying. Masses of 0.125–0.063 mm rocks were characterized by high humidity, and sensitivity to drying.

Taking into account the granulometric composition of the coal waste and based on the previously selected optimal fractional compositions of overburden rocks of different lithological types and horizons of occurrence, a series of samples—cylinders 50 mm in diameter and 45–55 mm in height—were molded. Optimal fractional compositions of ceramic charges are given in Table 5.

Table 5. Optimal fractional composition of overburden rocks of coal mining.

Material	Overburden Content, in % for the Grain Size, mm		
	0.5–0.25	0.25–0.125	0.125–0.0063
Argillite	5–45	10–55	15–50
Siltstone	10–55	15–50	15–45

The molded samples were kept in natural conditions for a day and were then dried in a desiccator for 24 h at a maximum temperature of 90 ± 5 °C. After drying, the samples were inspected and all changes in appearance were recorded, and air shrinkage and other characteristics were determined. The firing was performed in a laboratory furnace with automatic control of the firing mode with an average temperature rise rate of 1–3 deg/min, and held at the maximum temperature of 1.5–2 h according to the established mode at a temperature of 1000 °C.

3.2. Formation of Condensation Structure during the Drying of Ceramic Bricks

The drying process transformed the coagulation structure of the shaped product into the condensation structure of the dry product. The formation of the condensation structure took place when the products continuously changed size as the water was removed from them. The magnitude of stresses resulting from shrinkage deformations should not exceed the strength limit of the product. The main technological requirement for drying in the production of ceramics is the absence of cracks on the surface of products and distortion of their shape (warping). The formation of cracks is the main defect in drying. These defects are associated with deformations of the material due to uneven moisture and temperature fields. It was also established that the formation of cracks was caused by excessive pressure of moisture vapor inside the product with intense internal vaporization. The most dangerous in this case were the stresses caused by variations in moisture content [31–33].

When moisture evaporates from the surface, drying will occur over the entire volume of the product if the moisture moves from the center to the surface of the material. The movement of moisture in the products proceeds under the influence of various factors. Its intensity depends on the structure of the molded product, the form of the moisture connection with the mass, and the basic parameters of drying. The main parameters are temperature, relative humidity, and velocity of the coolant [34,35]. According to the relevant classification, with regard to the coefficient of sensitivity to drying (K_c), coal waste can be classified as a raw material with low susceptibility to drying. When exposed to heat flow, the time of cracking is 98–180 s, which is also characteristic of ra materials with susceptibility to drying. To determine the possibility of controlling the drying properties of coal mining wastes, studies of the dependence of changes in the condensation structure of samples on the fractional composition of rocks were conducted.

When planning and conducting experiments, the authors developed three-component fractional compositions in which the following particle-size intervals were taken as X_1, X_2, and X_3: X_1 = 0.5–0.25 mm; X_2 = 0.25–0.125 mm; $X_3 \leq$ 0.125 mm. Thus, both for argillites and siltstones, regardless of the horizon of overburden rock occurrence, grain sizes were conventionally designated as 0.5 mm, 0.25 mm, and 0.125 mm, and fractional compositions, from which the samples were formed, in each horizon of the coal waste were marked with serial numbers from 1 to 6 (Table 6).

Table 6. Marking of samples from coal-mine overburden.

Raw Material	Overburden at Horizons: +50 m; +100 m; +150 m					
	Argillite			Siltstone		
Particle size	0.5 mm	0.25 mm	0.125 mm	0.5 mm	0.25 mm	0.125 mm
Composition serial number	1	2	3	4	5	6

The results of the study of the drying properties of Ekibastuz coal basin overburden are shown in Table 7. Air shrinkage in argillites samples was 2.8–7.0%, and siltstones samples was 2.7–6.1%. With a change in the degree of rock crushing from 0.5 to 0.125 mm, an increase in shrinkage of 1.7–2.1-fold for argillites and 1.5–1.7-fold for siltstones was observed. A change in the rock horizon from +50 to −+150 m was characterized by a decrease in the coefficient of sensitivity to drying, air shrinkage, and an increase in the period of irradiation until the appearance of cracks. After analyzing the obtained data, we can conclude that the coal-mine waste showed the capacity for plastic formation when crushed to a grain size smaller than 0.5 mm and was moderately plastic and had low susceptibility to drying.

It was found that the reduction in shrinkage deformations was achieved by increasing the moisture conductivity and homogeneity of the structure of molded products. The kinetics of the drying process of the studied coal mining wastes was studied by determining the continuous change in shrinkage and mass of the samples. Figure 7 shows curves of dependence of shrinkage on the moisture of samples from coal mining wastes from different horizons of the optimal fractional composition. By analyzing the obtained curves, it can be noted that during the drying of samples, when capillary water was removed, shrinkage increased (region I), and when pore water was removed, shrinkage stabilized and became constant (region II). The inflection point of the curves corresponded to the moisture yield at which the shrinkage deformations were minimal. The moisture yield interval in the region I determined the critical moisture content of the samples (shrinkage completion).

Table 7. Drying properties of overburden rocks.

Composition Serial Number	Critical Humidity, Wcr, %	Susceptibility to Drying Ratio, $\frac{W_f - W_{cr}}{W_{cr}}$	Irradiation Period before Cracking, Z_o, s	Linear Air Shrinkage, %
Horizon +50 m				
1	7.8	1.31	129	3.8
2	8.9	1.41	125	5.6
3	8.8	1.44	103	7.0
4	8.8	0.91	121	3.3
5	9.8	1.02	105	5.4
6	10.2	1.09	98	6.1
Horizon +100 m				
1	8.2	1.11	151	3.2
2	8.5	1.21	140	5.1
3	9.4	1.31	128	6.3
4	8.8	0.84	135	3.2
5	9.8	0.89	121	5.1
6	10.2	0.94	107	5.7
Horizon +150 m				
1	7.3	1.03	159	2.8
2	8.4	1.12	151	4.7
3	9.1	1.25	141	5.5
4	8.6	0.77	147	2.7
5	9.5	0.82	129	4.3
6	10.2	0.88	125	5.0

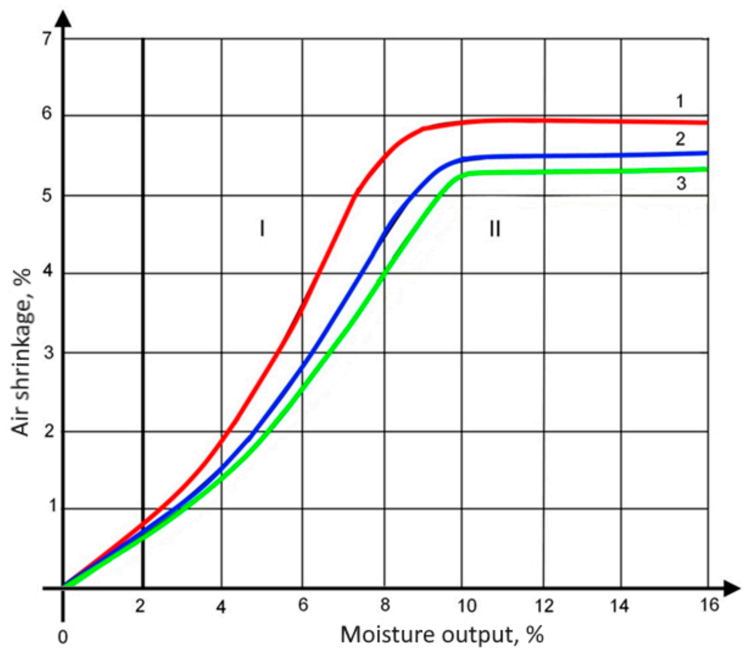


Figure 7. Dependence of air shrinkage on the moisture content of samples from the coal-mine waste of the optimal fractional composition: 1: argillite of +50 m horizon; 2: argillite of +100 m horizon; 3: argillite of +150 m horizon.

For coal-mine waste horizon +50 m with clay mineral content of 20–55%, the area of stabilization of shrinkage deformation began at the moisture content of 8.4%. For coal-mine waste horizons +100 m and +150 m, which were characterized by clay mineral content ranging from 20 to 45%, the completion of shrinkage occurred at the moisture content of 9.6 and 10.1%. An analysis of the obtained results (Tables 8 and 9) showed that as

0.125 mm particle content increased up to 50%, and 0.5 mm particle content up to 30%, a 4–5% decrease in air shrinkage of samples was observed. By drying sensitivity rate, the masses from coal-mine waste belonged to the low sensitivity variety. It was shown that when limiting the content of 0.125 mm particle content to 45–70% in the process of drying, shrinkage deformation of samples decreased. Taking into account the results of studies of coagulation structures of argillite–siltstone masses, the optimum proportions of particle sizes providing the formation of defect-free condensation structures were selected (Figure 6). Furthermore, the value of the Wcr/Wf ratio decreased from 0.5–0.6 to 0.3–0.4 on average, which was indicative of a more rapid achievement of the constant shrinkage interval during drying.

Table 8. Dependence of physical and mechanical properties of dried samples on the fractional composition of coal mining waste.

Composition Number	Forming Moisture, W_{rel}	Critical Humidity, W_{cr}	Air Shrinkage, %	Apparent Porosity, %	Fracture Strength, MPa
		Horizon +50 m			
1	17.4	8.0	4.3	10.0	1.8
2	18.2	5.1	2.3	16.5	1.4
3	18.5	4.7	2.4	16.4	1.1
4	17.8	7.4	3.7	14.6	1.8
5	19.3	7.6	3.6	14.8	2.1
6	18.4	5.2	2.2	16.6	1.4
		Horizon +100 m			
1	17.6	7.2	3.3	13.1	3.3
2	18.2	3.2	0.8	16.2	1.6
3	18.6	2.2	0.7	18.0	1.2
4	18.4	5.3	2.3	14.5	2.8
5	18.9	4.9	2.4	14.0	2.0
6	18.5	2.6	0.8	15.9	1.5
		Horizon +150 m			
1	16.8	8.7	5.8	19.8	2.6
2	17.4	8.1	3.4	10.9	2.2
3	18.2	4.1	2.4	12.4	3.3
4	17.6	8.0	4.5	18.3	3.1
5	17.8	6.6	3.9	19.9	2.1
6	18.2	4.4	2.3	10.7	3.1

Table 9. Physical and mechanical properties of dried samples from coal-mining waste of optimal fractional composition.

Molding Conditions on the Press	Air Shrinkage, %	Breaking Strength, MPA	Apparent Porosity, %	Gas Permeability, K_a 10^{-15} m^2	Apparent Density, kg/m^3
		Horizon +50 m			
without vacuum	2.5	1.9	15.9	18.1	1840
vacuum	1.6	3.5	14.3	1.4	1833
		Horizon +100 m			
without vacuum	1.1	2.1	16.0	24.4	1898
vacuum	0.8	2.8	15.4	2.0	1835
		Horizon +150 m			
without vacuum	3.4	1.9	9.9	8.1	1869
vacuum	3.0	3.4	9.7	1.1	1828

The decrease in molding moisture and air shrinkage occurred due to a decrease in the content of the 0.125 finely dispersed coal-mining waste. A decrease in the ratio W_{cr}/W_f was caused by an increase in the share of pore water in the mass, which was confirmed by an increase in the apparent porosity of samples by 2–3%.

Thus, changing the fractional composition of crushed coal mining waste made it possible to control not only the drying properties but also the gas permeability of samples, which is an important condition for the burning of organic matter during firing [36]. The increase of 0.5 mm particle-size content up to 30–50% and 0.25 mm particle-size up to 25–30% in the crushed coal mining wastes resulted in an increase in gas permeability of samples, in comparison with the samples with a predominant 0.125 mm particle size. In the space occupied by these compositions, the densest packing of particles of crushed coal mining waste occurred.

Taking into account the results of the research, the previously limited areas of fractional compositions of coal-mining waste providing the formation of ceramic masses with optimal molding properties were adjusted (Figure 8).

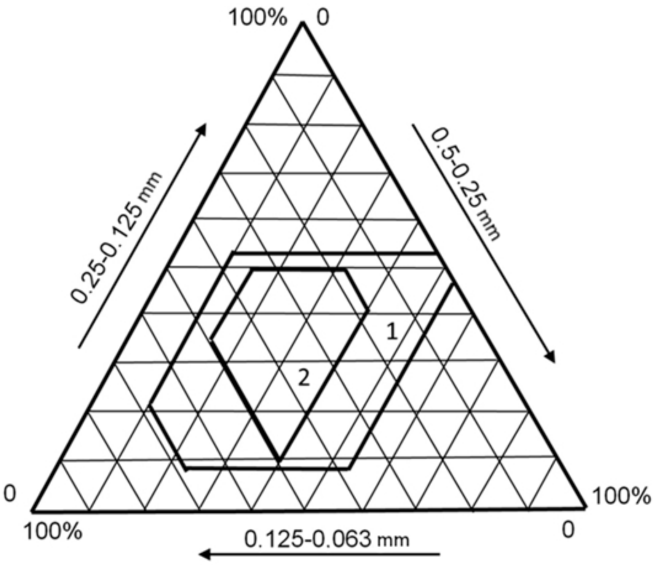


Figure 8. Diagram of the fractional composition of overburden rocks of coal mining waste optimal in structural–mechanical (1) and drying (2) properties.

The homogeneity of the dried product structure was determined by the change of apparent density along the length of the product, which varied by 1–3%. It should be noted that when forming samples using vacuumization, the fracture strength of dried products was 1.5–2 times higher due to the compaction of products. At the same time, gas permeability of samples decreased to a greater extent than apparent porosity, as a result of changes in the number and shape of pores.

3.3. Formation of Crystallization Structure during the Firing of Ceramic Bricks

The firing of ceramic products was the main limiting factor affecting the formation of the crystal structure, physical and mechanical properties, and quality of the finished products.

Coal-mining waste mineral composition belonged to the kaolinite–hydrosludite raw materials and differed from the traditional clays in that it contained organic matter. Transformations processes during firing were mainly studied for clay-based systems, and changes in the phase composition of coal mining wastes at different firing temperatures were insufficiently studied. To optimize the firing regime and to study the process of mineral formation in conditions of intense burning of organic matter, study on determining melting characteristics, as well as differential-thermal, petrographic, and diffractometric analyses of samples fired at different temperatures, were conducted [37].

In the MNO-2 heating microscope, the temperatures corresponding to the stages of deformation of samples during their heat treatment were set as follows: T_1—temperature of the beginning of deformation of samples; $T_{m.y.}$—temperature of maximum shrinkage of samples; $T_{m.v.}$—temperature of maximum swelling of the samples; T_2—softening temperature, at which the sample formed a hemisphere; T_3—temperature of liquid state. The results of the determination are provided in Table 10.

Table 10. Fusibility characteristics of coal-mine overburden.

Name	T_1, °C	$T_{m.y.}$, °C	$T_{m.v.}$, °C	T_1, °C	T_3, °C	Sintering Interval
			Horizon +100 m			
Argillite	1050	1250	1470	1500	1550	220
Siltstone	1000	1240	1470	1580	1590	230

An analysis of the obtained data (Figure 9) showed that the beginning of the deformation of samples from argillites was observed at 1000–1080 °C with an increase in the specified temperature from horizon +150 m to horizon +50 m.

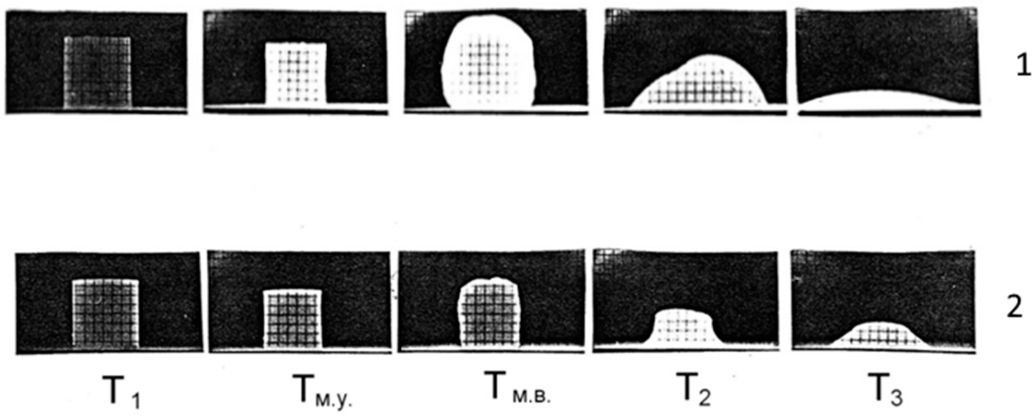


Figure 9. Melting characteristics of the samples: 1—argillite, 2—siltstone.

The beginning of the sintering of samples was recorded at 1240–1250 °C, and the sintering interval was 210–230 °C. Based on their sintering temperature of 1240–1250 °C, the rocks in question can be classified as the medium-temperature sintering type. At 1450–1470 °C, the bloating of samples was observed. The softening temperature, at which the sample turned into a hemisphere (T_2), can be considered as an indicator of the refractoriness of rocks because at this temperature, the melting of samples did not occur yet. Argillites belonged to the group of refractory raw materials with a refractoriness index of 1500–1580 °C. The liquid-melting state of argillites corresponded to the temperature range of 1550–1590 °C.

The above lead us to conclude that it is advisable to fire products from argillites to the maximum temperature of approximately 975–1025 °C under the conditions of organic substances burning with the formation of minerals contributes to the formation of ceramic shards. The firing of samples from coal mining wastes of optimal fractional composition was carried out to the maximum temperatures of 950, 975, and 1000 °C, with a firing interval of 100 deg/h, while studying the processes of mineral formation (Table 11).

The samples burnt at 450 °C contained unchanged quartz, feldspars, iron oxides and hydroxides, and clay minerals. In appearance, the samples had a black color in the middle and dark red at the edges, indicating the beginning of burnout of organic matter. Diffractograms patterns confirmed the presence of quartz, feldspars, and clay minerals represented by kaolinite, hydromica, and montmorillonite. The reflexes of kaolinite disappeared on the diffractograms images at 550–800 °C, which indicates the dehydration of clay minerals and the transition of kaolinite to meta kaolinite. Under the microscope, melted quartz grains in the marginal zone of the samples were observed. The samples acquired a denser structure in the light marginal zone. The central part of the samples had a dark zone with unburned organic matter.

Table 11. Temperature ranges of physical and chemical processes.

Basic Physicochemical Changes	Temperature, °C		
	Argillite		
	Horizon +50 m	*Horizon +100 m*	*Horizon +150 m*
Low-temperature water extraction	90–100	90–190	90–200
Ignition and combustion of volatile combustible components	250–380	250–380	270–390
Medium-temperature constitutional water extraction	540–620	480–630	520–630
Ignition and combustion of organic matter	430–700	450–780	450–790
High-temperature water extraction	760–800	720–810	740–800
Combustion of organic matter	850–950	910–950	920–975
Start of liquid phase formation	860	910	930
Crystallization of neoplasms involving the liquid phase	960–990	970–990	980–1000

At a firing temperature of 900 °C, the clay minerals amorphized and the liquid phase appeared. Diffractograms patterns showed reflexes of quartz, feldspar, and hematite. At 950 °C, the samples constituted burned shards with a burnt zone, but there was a small central dark-colored zone with unburned organic matter. The quartz and feldspar grains were melted. On diffractograms, reflexes of quartz (d/n = 0.425; 0.334; 0.228; 0.212 nm) and feldspar (d/n = 0.403; 0.323; 0.319 nm) persisted. Reflexes of hematite (d/n = 0.269; 0.208 nm;) and cristobalite (d/n = 0.251 nm) appeared. Increasing the firing temperature to 975 °C lead to further thickening of the shard structure, accompanied by an increase in the amount of liquid phase. The organic matter was completely burned out and the black core disappeared. Melted quartz and feldspar grains were observed, and mullite crystals were present in the light brown areas. Reflexes of quartz, feldspar, hematite, mullite, and cristobalite were also recorded on the diffractograms.

When the samples were fired up to 1000 °C, complete amorphization of clay minerals occurred, accompanied by a further increase in the liquid phase and compaction of the shards with a decrease in the linear dimensions of the samples. The marginal zone of samples had a light brown color, but the core of the dark color remained unburned. This was due to intensive sintering of the marginal zone and in the central zone due to limited oxygen access, whereby the organic matter did not have time to burn out. Reflections of quartz, feldspar, hematite, mullite, and cristobalite were preserved on the diffractograms (Figure 10).

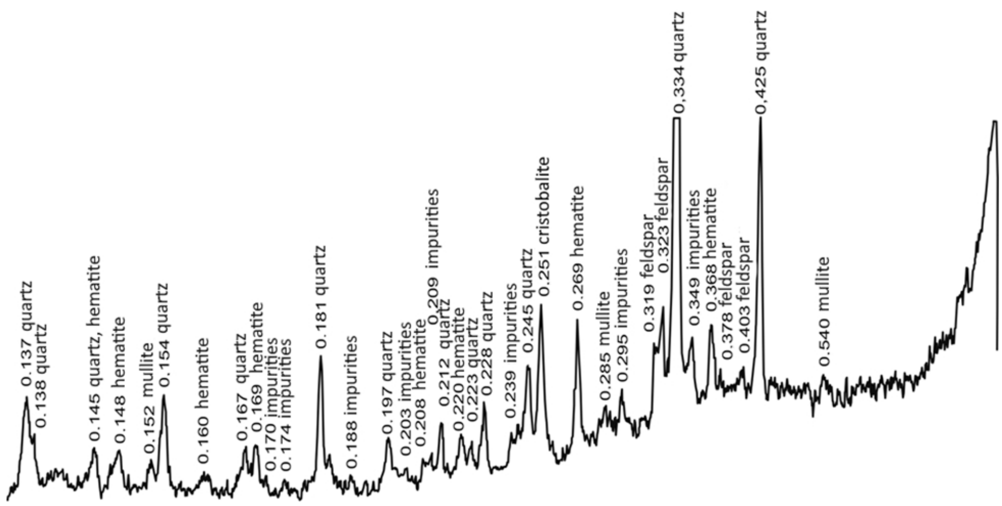


Figure 10. Diffractogram of coal-mine overburden rocks, argillites, after firing at 1000 °C.

Petrographic and diffractometric studies of samples from coal-mine waste of various horizons showed that starting from the temperature of 950 °C, intensive amorphization of clay minerals was observed, with an increase in the amount of liquid phase, linking relicts of quartz and ore minerals (Figure 11).

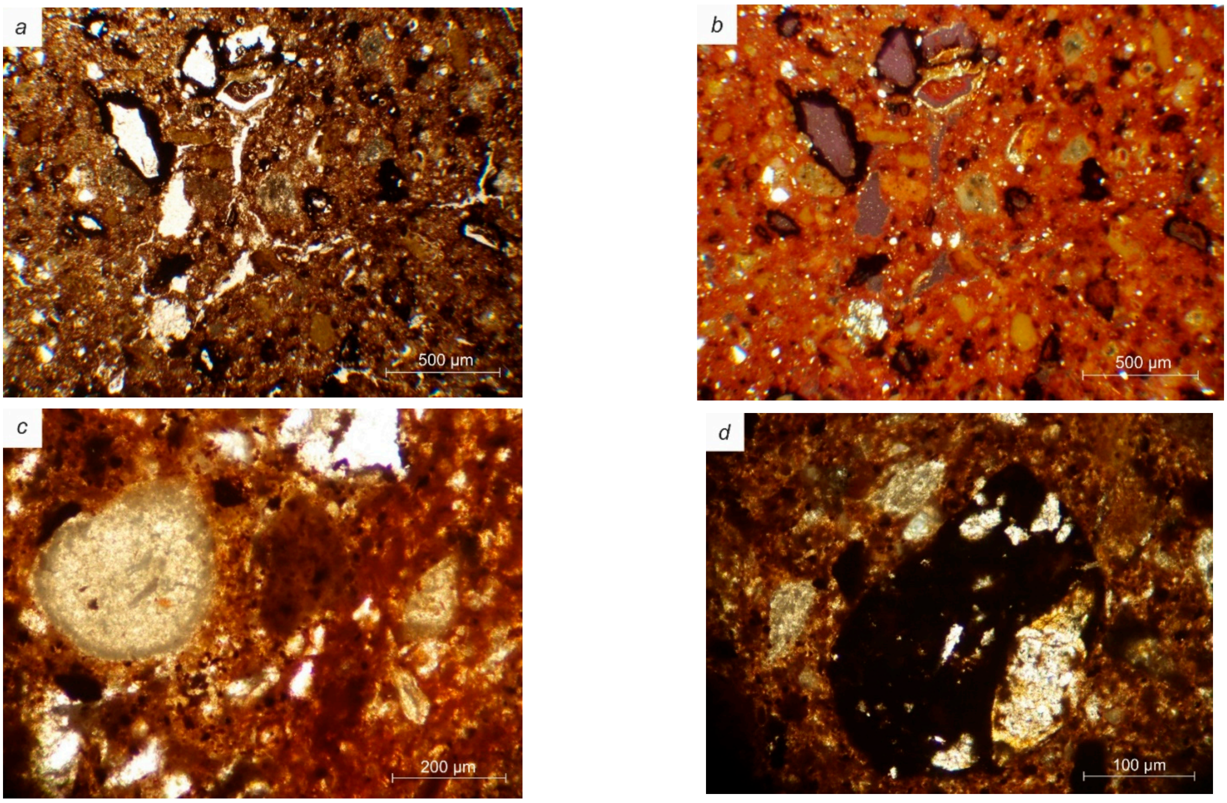


Figure 11. Microphotographs of the structure of argillites fired at 1000 °C. Thin section, transmitted light: 25×, nicoli II (**a**); 25×, nicoli + (**b**); 75×, nicoli II (**c**); 150×, nicoli + (**d**).

Based on the studies, the temperature rise rate of 100 deg/h, up to 800 °C, and 20 deg/h, up to a maximum temperature of 1000 °C, was accepted.

To determine the maximum temperature and the duration of isothermal soaking at the maximum temperature, we studied the change in physical and mechanical properties of ceramic products from coal-mine waste, fired in three modes. The maximum firing temperature was adjusted within the range of 950–1000 °C. The duration of isothermal soaking at the maximum firing temperature was 2 h.

At a firing temperature of 1000 °C, there was a complete burning out of organic matter and the disappearance of the black core. The process of recrystallization of clay minerals with the formation of new crystalline phases of hematite, mullite, and cristobalite was completed, which provided optimal physical and mechanical properties of finished products from coal-mine waste (Table 12).

Physical and mechanical tests of the fired samples showed that the highest strength characteristics were exhibited by the samples fired up to 1000 °C. Frost resistance was 35–75 cycles of alternate freezing and thawing without signs of destruction (peeling and flaking). A distinctive feature of the firing protocol was a slowdown in the rate of product heating in the range of burnout of organic matter (800–975 °C). The maximum firing temperature was 1000 °C, and the duration of isothermal exposure was 2 h.

Table 12. Physical and mechanical properties of fired samples.

Raw Material	Max. Firing Temperature, °C	Total Shrinkage, %	Strength in Compression, MPa	Water Absorption, %	Frost Resistance, Cycles
Horizon +100 m					
Argillite	950	4.50	22	12.3	50
	975	4.62	28	11.5	75
	1000	4.80	31	8.6	75
Siltstone	950	3.60	19	12.4	35
	975	3.72	23	11.2	50
	1000	3.81	26	10.8	75

3.4. Effect of Organic and Chemical Additives on Quality Indicators of Ceramic Bricks from Coal-Mine Overburden

Control of the structure and properties of the mass during their processing can be achieved by introducing various additives that help change the structural and mechanical properties of the mass within a wide range. Organic and plasticizing additives have a positive effect on the molding and structural-mechanical properties of the masses.

The influence of highly mineralized coal-bearing rocks and highly basic technical-grade aluminum oxychloride on the qualitative indices of products from coal-mine overburden was studied.

Highly mineralized carbonaceous rocks were previously ground to a particle size smaller than 0.125 mm and introduced into mixtures in an amount of 4–12% (compositions 1–3, Table 13).

Table 13. Raw mix compositions containing additives.

Components	Content of Components in the Mixture, Mass %					
	1	2	3	4	5	6
Overburden rock from coal mining	96	92	90	97	95	93
Highly mineralized carbonaceous rocks with 3–9% resinous and bituminous substance content	4	8	12			
Highly basic technical aluminum oxychloride				3	5	7

The mechanism of the impact of carbonaceous rocks on the rheological properties of the masses was that the resin and bitumen substances present in these rocks gave rise to coagulation phenomena in the masses and provided a homogeneous and more plastic mass due to the ability of the clay components of siltstone–argillite rocks to emulsify "resin-water" and "bitumen-water" systems as a result of wetting and sticking [38,39].

The introduction of carbonaceous rocks containing resin and bituminous substances reduced the plastic viscosity of the masses by 6–13%, which improved the mobility of the masses, reduced the plastic strength by 18–23%, which led to a decrease in rigidity and had a favorable effect on the molding ability of the masses [40,41].

The use of highly mineralized carbonaceous rocks allowed for the enhancement of molding properties of the masses, a reduction in water absorption of products, as well as an increase in their physical and mechanical properties (Table 13).

As a liquid additive to improve structural and mechanical properties of overburden mass, a highly basic technical aluminum oxychloride was used, which was obtained by dissolving anhydrous aluminum chloride sludge in water, with subsequent neutralization by hydrochloric acid formed in the solution as a result of hydrolysis of the latter, an interaction with a metal oxidizer. Aluminum oxychloride composition corresponded to the formula $[Al(CH)_3\text{-}XClX]_n$, where X = 0.5; n = 2.

Raw mixes of overburden crushed to particles smaller than 0.5 mm were moistened with water mixed with aluminum oxychloride in an amount of 3–7%, to normal molding moisture (composition 4–6, Table 13).

When studying the effect of aluminum oxychloride on the structural and mechanical properties of overburden masses, it was established that the mechanism of the aluminum oxychloride effect was identical for all lithological rock types and did not depend on the depth of their occurrence.

The aluminum oxychloride inclusion helped to improve the molding and rheological properties of the masses by reducing their plastic viscosity by 40% and plasticity strength by 20–25%. Indexes of plasticity of these masses 0.48–0.66 were close to values typical for well-forming masses [42–46]. There was a redistribution in the ratio of deformations towards the prevailing development of deformations, which indicates that the masses belonged to the first structural-mechanical type.

The results of determining the physical and mechanical properties of the obtained products are shown in Table 14.

Table 14. Results of physical and mechanical tests.

Composition Number	Component Content in Mass, %			Compressive Strength, MPa	Water Absorption, %	Frost Resistance, Cycles
	Overburden	Eelgrass	Aluminum Oxychloride			
1	96	4		42.0	10.8	58
2	92	8		48.2	9.2	72
3	88	12		44.0	9.6	74
4	97		3	34.2	8.2	62
5	95		5	33.6	7.4	63
6	93		7	36.4	7.0	68

The influence of aluminum oxychloride on the physical and mechanical parameters of the obtained products was manifested in the reduction in water absorption from 8.2 to 7.0%, and an increase in compressive strength by 10–12%. Products containing aluminum oxychloride can withstand 75 cycles of alternate freezing and thawing and have a good appearance.

4. Conclusions

Based on the outcomes of the research on processes of structural formation, ideas for producing composite ceramic articles using coal-mine overburden were elaborated.

Overburden rocks from coal mines, as far as their physical and mechanical, technological properties, chemical and mineral composition are concerned, are close to the traditional clay raw materials and can be used for the production of ceramic products, provided that appropriate technological preparation is ensured.

It was established that by virtue of having a high degree of metamorphism, overburden is not soaked in water and exhibits the capacity for plastic formation when their condensation and cementation bonds are destroyed by grinding to a particle size of less than 0.5 mm. The crushed rocks are moderately plastic and have low sensitivity to drying.

The control of fractional composition of coal mining wastes leads to an improvement of molding and drying properties of ceramic products derived from them. We determined the range of particle sizes from coal-mine overburden with optimal molding and drying properties: 0.5–0.25 mm: 5–50%; 0.25–0.125 mm: 5–60%; under 0.125 mm: 45–70%.

Samples of the optimized coal mining wastes molded on the vacuum press exhibited air shrinkage of 0.8–3.0%, ultimate fracture strength of 2.3–3.3 MPa, apparent porosity of 8.7–15.4%, and density of 1828–1990 kg/m^2.

The improvement of structural and mechanical characteristics of masses from coal-mine overburden rocks was achieved by using highly mineralized carbonaceous rocks and aluminum oxychloride as organic and plasticizing additives. The products fabricated with the use of additives had a compressive strength of 33.6–48.2 MPa, frost resistance of 50–75 cycles, and water absorption at 7.0–10.8%.

Using the research results, the following optimal firing regime was adopted: temperature rise rate of 100 deg/h, up to 800 °C, and 20 deg/h, up to a maximum temperature of 1000 °C. The duration of isothermal soaking at the maximum temperature was 2 h.

Energy-efficient, resource-saving technology for the production of composite ceramic products using coal-mine overburden will significantly reduce the cost of bricks, compensate for the lack of high-quality clay raw materials and improve the environmental situation. The results obtained warrant further research.

Author Contributions: Conceptualization: V.S. and A.S.; methodology: G.R.; software: M.R.; validation: V.S., A.S. and M.R.; formal analysis: V.S., A.S. and M.R.; investigation: A.S. and O.V.; resources: M.R. and V.S.; data curation: A.S. and P.K.; writing—original draft preparation: O.V. and A.S.; writing—review and editing: G.R. and O.V.; visualization: O.V. and A.S.; supervision P.K.; project administration G.R.; funding acquisition: M.R. and V.S. All authors have read and agreed to the published version of the manuscript.

Funding: This research did not receive external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Informed consent was obtained from all subjects participating in the study.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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