

Overburden coal output rocks as a raw material for making ceramic building materials

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Abstract. The relevance of this study is conditioned by the need to solve environmental, economic, and engineering problems of disposing of coal mining overburden rocks from the Ekibastuz coal basin. This article analyzes the properties of coal mining overburden rocks and the possibility of producing ceramic building materials on their basis. The features of the stones depending on their lithological type and occurrence horizons are identified during a set of electronic microscopic, X-ray phase, petrographical, and chemical tests conducted by modern test methods. This work studies the physico-mechanical properties and the chemical and mineralogical composition of coal mining overburden rocks. It reveals their similarity to conventional argillous raw materials used for producing ceramic building materials. The content of the article is of practical value for both, coal industry enterprises producing overburden rocks as production wastes and for enterprises producing ceramic building materials. These materials can significantly reduce the prime cost of manufactured products and fill the lack of on-spec argillous raw materials when using coal mining overburden rocks.

Keywords: ceramic materials; chemical composition; overburden rocks; strength

1. Introduction

The open coal mining at the world's largest deposit in Ekibastuz results in the formation of a huge amount of percarbonic mass, that is, overburdened rocks removed to open dumps. The currently accumulated amount of coal mining overburden rocks is about 4 billion cubic meters. Overburden rock dumps occupy vast areas and reach up to 100 m in height. They significantly aggravate the environmental conditions in the region, and cause dust storms, ignitions, and gas contamination.

Coal mining overburden rocks differ from other kinds of mineral raw materials by organic matter

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content and physico-mechanical and physico-chemical properties conditioned by their formation during carbonification and metamorphization (Kotlyar *et al.* 2017, Stolboushkin *et al.* 2016, Storozhenko *et al.* 2010, Vereshchagin *et al.* 2016,).

The large-scale utilization of coal waste is impeded by its understudied composition, homogeneity, and technical properties, the absence of any procedures for their evaluation, and scientifically underpinned coal waste processing technologies (Abdrakhimova *et al.* 2017, Vasić *et al.* 2021, Yavruyan *et al.* 2018).

Coal mining waste is a promising basic raw material for producing ceramic building materials. Currently, the most broadly used technology consists of utilizing coal mining waste as additives for producing ceramic goods (Mishra *et al.* 2023a, Gaishun *et al.* 2020, Yavruyan *et al.* 2019, Stanevich *et al.* 2022, Tkach *et al.* 2018, Akishev *et al.* 2024, Rakhimova *et al.* 2024). The purpose of this work is the comprehensive study of coal mining overburden rocks as the main raw materials for making efficient wall and decorative ceramic products. The objectives of this study are to perform petrographical tests and study the physico-mechanical properties and chemical and mineralogical composition of coal mining overburden rocks.

2. Materials and methods

The overburden rocks, that is, the coal mining waste from the Ekibastuz coal basin that are represented by argillites, aleurolites, and intercalations are valuable organic mineral raw materials and can be used as the main raw materials for producing ceramic building materials. Some of the physico-mechanical properties of overburden rocks depend on the level of their metamorphization. In their natural form, these rocks do not swell on wetting, which requires exposing them to mechanical grinding to destroy cementation bonds among clay components (Girnis *et al.* 2024, Bulyga *et al.* 2024, Ryzhkov *et al.* 2021).

The chemical composition of the overburdened rocks was determined according to GOST 2059-95 (Solid Mineral Fuel, 2002). Their physico-mechanical properties were determined according to GOST 7025-91 and GOST R 58527-2019 (Ceramic and calcium silicate bricks and stones, 2006, Wall materials, 2019).

The petrographical tests were conducted on semi-translucent polished sections with the help of a Polmi-A microscope. Those specimens were made by conventional methods. The mineralogical phase composition of the rocks was analyzed using an X-ray diffraction meter DRON-3.0. Because coal mining overburden rocks contain organic matter, their handling properties were determined by conventional procedures for argillous materials and according to GOST 21216.1 – 21216.5-81 and GOST 21216.9 – 21216.11281.

3. Results and discussion

As shown by the macroscopic description of the overburdened rocks, they were mainly dense and solid and their color varied from light gray to dark gray due to the uneven saturation with organic matter (Table 1, Fig. 1). The rocks also contain finely dispersed carbonates the processing of which using 10% hydrochloric acid results in minor boiling up. As shown by the petrographic tests of the overburdened rock specimens, they consisted mainly of argillous and aleurite materials the fraction of which reached 55-90%. The mineral impurities in the overburdened rocks included quartz, feldspar, mica, and pyrite. Their fraction ranges from 1 to 5%. The main amount of the

Table 1 Macroscopic description of overburden rocks

Lithological type of raw materials	Brief description of raw materials	Texture	Structure	Sample in the interaction with 10
Aleurolite	Light to light gray, dense, solid	Microlamellar structure conditioned	Phytohemoleuritic, with psammitic elements	Slight boiling up
Argillite	Light gray to dark gray, dense, solid	Directional structure conditioned by various sandy/siltstone material	Phytohemoaleuropelitic	Slight boiling up

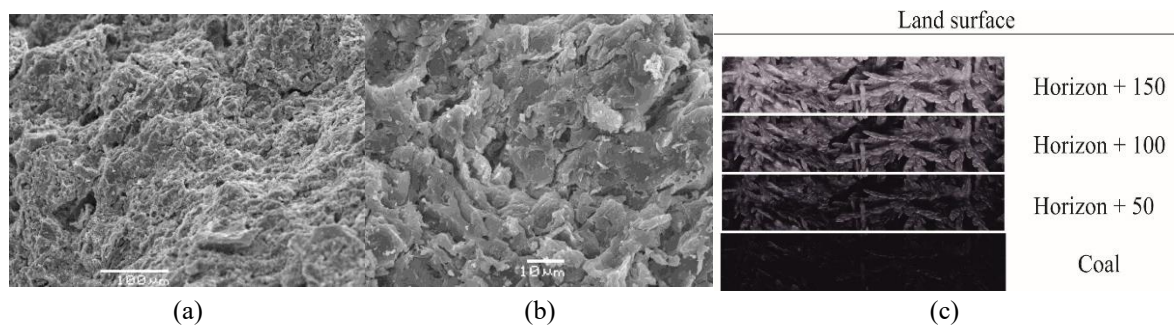


Fig. 1 Structure of polished sections of aleurolite (a), argillite (b), horizons (c)

organic matter permeating the argillous part of the rocks was represented by carbonified residues of plant tissues. The content of various organic compounds decreases from argillite to intercalations and aerolite.

The minerals with a density below 2.9 g/cm³ are quartz, feldspar, rock fragments, chlorites, and carbonified organic residues. The minerals of this fraction constitute the groundmass.

The minerals with a density exceeding 2.9 g/cm³ include the loadstone-titanium ferrite group, bloodstone-limonite group, leucoxene, zircon, garnet, tourmaline, andalusite-sillimanite group, hematite group, zoisite, amphibole, monoclinic pyroxenes. The autogenous heavy cuts contained in the overburdened rocks include barite and pyrite.

The light cuts of aleurolite are represented by a higher content of quartz (19.2 %) and rock fragments (53.1 %) than in argillite. There is also a small fraction of feldspar (2.6 %). However, the organic matter content is only 25.2 %, which is a much lower percentage than in argillite.

The light cuts of aleurolites differ from the light cuts of argillites by a more depleted association of minerals. Aleurolites also contain a much smaller amount of bloodstone-limonite (4.8 %) and loadstone-titanium ferrite but a lot of leucoxene (43.6 %). The auxiliary minerals in aerolite include rutile, tourmaline, and others. Aleurolites also contain some autogenous minerals, including a lot of barite (38.8 %) and a much higher percentage of pyrite (4.7 %) than argillite. The carbonate content is 6.5 %.

The light fraction of argillite is represented by quartz (13.2 %), rock fragments (38.2%), and organic matter (48.6 %). The quartz grains are colorless, often contaminated with various impurities, round, and, more often, irregularly shaped. The grain contains mineral impurities and coal dust. Rock fragments are represented mainly by siliceous and quartz siliceous rocks. Autogenous minerals are represented by calcitic grains. Heavy argillite cuts are represented by diverse minerals the main of which by content are bloodstone limonite (49.9 %) and loadstone with titanite iron ore (13.6 %).

Table 2 Physico-chemical properties of overburden rocks

Raw material	Density, g/cm ³	Natural moisture content	Compression strength, mPa	Porosity, %
At +50 m horizon				
argillite	2.4	5.1	37.4	12.9
aleurolite	2.7	4.3	48.9	10.6
At +100 m horizon				
argillite	2.4	4.9	34.9	14.9
aleurolite	1.5	4.3	47.0	15.9
At +150 m horizon				
argillite	2.2	5.8	34.7	18.7
aleurolite	2.3	4.7	45.0	19.1

The autogenous minerals contained in the overburden rocks are barite (16.1%) and pyrite (0.8%), whereas the carbonate content is 7.5%. Auxiliary minerals are represented by garnet, hematite, tourmaline, rutile, and others, whereas the quartz content in light intercalation cuts is 13.9 %. The rock fragments content is 48.6 %; the organic matter content is 33.2 %. Groups of micaceous minerals are also detected, including biotite, muscovite, and chlorite; heavy intercalation cuts are represented by a diverse set of clastic minerals, auxiliary minerals included; however, their set is more depleted than in argillite and aerolite. The detected fractions of leucoxene, loadstone titanoferrite, and bloodstone limonite are more significant and amount, respectively, to 39,2 %, 11.4 %, and 10.1 %. The respective barite and pyrite content in autogenous heavy minerals is 27.6 % and 8.5 % pyrite.

The quality of ready-made ceramic products of coal mining waste is heavily influenced by the physico-mechanical properties and chemical mineralogical composition of overburden rocks. The occurrence horizons of the overburdened rocks (argillites and aleurolites) covered in the studies were +50, +100, and +150 m. Horizons are taken from the top of the coal seam, Fig. 1.

The main physico-mechanical properties of overburden rocks are density, natural moisture content, compressive strength, and porosity (Table 2). According to the density analysis of the study results, the overburden rock density gradually decreases with the transition from the +50 m to the +150 m horizon. In this case, the density of aleurolites is 7-10 % higher than the density of argillites. The respective densities of argillites and light-gray argillites are 2.3-2.7 g/cm³ and 2.6 g/cm³. The respective natural moisture content in argillites and aleurolites ranges from 4.8 to 5.8 %, and from 4.3-4.7% and rises with the transition from the +50 m to the +150 m horizon. The natural moisture content in light-gray argillites was 4.8%. The respective compressive strengths of overburden rocks were 34.7-37.4 mPa for argillites, 45 to 48.9 mPa for aleurolites, and 45.8 mPa for light-gray argillites. The change in the occurrence horizon from +50 to +150 m is characterized by the reduction in the compressive strength of aleurolites and argillites. The respective porosity of argillites and aleurolites ranges from 12.9 to 18.7% and from 10.8 to 19.1% and increases with the transition from the +50 m to +150 m occurrence horizon. The porosity of light-gray argillite is 15.6 %.

Thus the change in the overburden rock occurrence horizons from +50 m to +150 m downgrades the physico-mechanical properties of the rocks, which stems from the reduction in the crumbling level of the rocks with this change in the horizons and their more significant weathering at the +150 m horizon.

Table 3 Chemical composition of the test samples

Raw material	Oxides, %											
	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃ +FeO	MgO	MnO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	SO ₃	l.o.i.
At +50 m horizon												
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
aleurolite	61.1	1.03	18.5	3.4	0.9	0.12	0.85	1.32	2.06	0.25	0.21	10.4
At +100 m horizon												
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
aleurolite	59.9	0.93	17.96	4.18	1.1	0.09	0.74	1.06	2.24	0.25	0.22	10.9
At +150 m horizon												
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
aleurolite	61.3	0.93	17.6	4.02	0.03	0.08	0.56	1.38	2.11	0.28	0.28	10.06

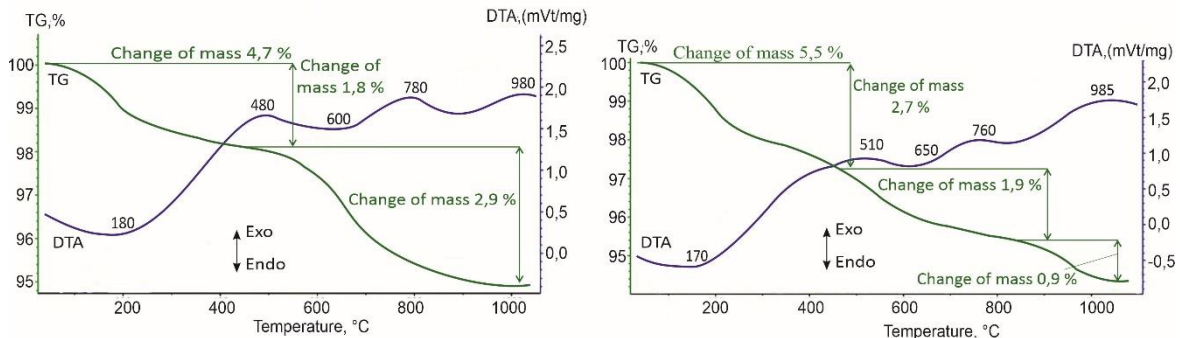


Fig. 2 Thermograms of argillite (a) and aleurolite (b), horizon +100 m

If to consider the data from the chemical analysis of overburdened rocks of various lithological types, it will be possible to calculate the quantitative content of rock-shaping oxides (Table 3).

The content of SiO₂ in the samples ranges from 56.7 to 61.3%. It is detected in free and bound states. Bound silica is contained in clay-forming minerals, whereas free silica is represented by finely dispersed quartz impurities, and its content in the samples ranges from 16 % to 25%. In terms of free quartz content, coal mining waste is considered raw materials with a medium quartz content. Aluminum oxide Al₂O₃ is contained in clay-forming minerals and micaceous impurities. The respective content of Al₂O₃ in argillites, aleurolites, and light-gray argillites ranges from 17.6 to 16.4%, from 17.6 to 18.5%, and 17.9% and decreases in argillites depending on the occurrence horizon (from +50 m to +150 m). In terms of calcined aluminum content, the rocks are classified as semiacid raw materials.

The respective content of ferrous oxides in the test samples ranges from 5.6 to 6.4% for argillites and from 3.4 to 4.18% for aleurolites. Ferrous compounds are represented by pyrite and siderite. In terms of ferrous oxide content, overburdened rocks are considered raw materials with a high content of coloring oxides. Alkaline earth metal oxides are contained in argillous minerals and carbonates. The aggregate respective content of calcium and magnesium in argillites and aleurolites is 1.67-2.3% and 1.09-1.84%.

The aggregate content of sodium and potassium oxides in the lithological types from various occurrence horizons ranges from 2.96 to 3.36%. Alkaline oxides are contained in clay-forming

Table 4 Mineralogical composition of overburden rocks

Raw material	Mineral content, %							
	Clay minerals		quartz	feldspar	carbonates	micas	siderite	Organic matter
	kaolinite	Hydrous mica						
At +50 m horizon								
argillite	25-30	20	25	8-12	-	-	-	15
aleurolite	5-10	25	30	20-25	3-5	5	-	10
At +100 m horizon								
argillite	25	20	30	-	-	-	5	12
aleurolite	5-7	20	40	25	3-5	5	-	8
At +150 m horizon								
argillite	15	20	40	10	-	-	-	10
aleurolite	3-5	20	50	15-20	5	-	-	5

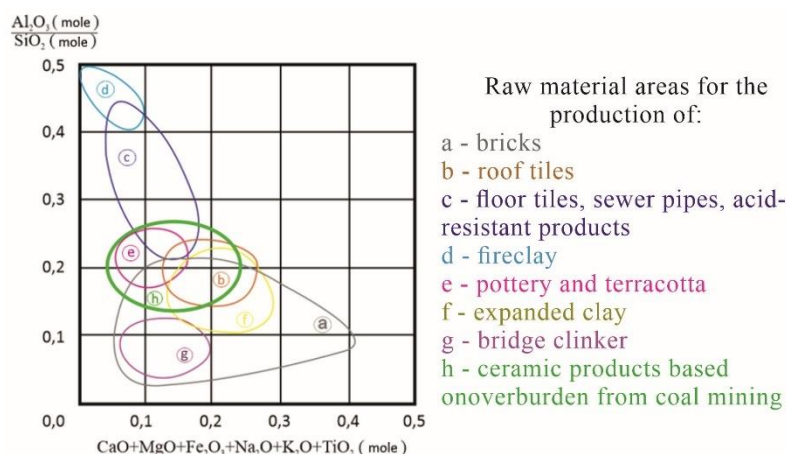


Fig. 3 Diagram of industrial purpose of clays depending on their chemical composition (A.I. Avgustinnik)

minerals and as water-soluble salts in impurities. The content of SO_3 does not exceed 0.28%, which is typical of eco-friendly raw materials with a low content of sulfur and allows using overburden rocks in manufacturing ceramic products without limitations. In addition, overburden rocks contain organic carbon.

The qualitative composition of argillites according to the data of differential thermal analysis is represented by hydromica and kaolinite (40-50%), quartz up to 30%, mixed-layer minerals, and organic matter.

The siltstones are characterized by the predominance of hydromica and quartz (20-50%), a group of mixed-layer minerals, and insignificant content of montmorillonite and organic matter.

Mineralogical composition of the studied overburden rocks depending on lithology and bedding horizon is presented in Table 4.

The mineralogical composition of overburden rocks is the kaolinite-hydrologic raw material with an admixture of quartz, feldspar, mixed layer minerals, and organic substances and can be used as the main raw material for the production of ceramic building materials. In terms of mineralogical composition, overburden (Vyshar *et al.* 2023, Rakhimova *et al.* 2023, Maussymbayeva *et al.* 2024).

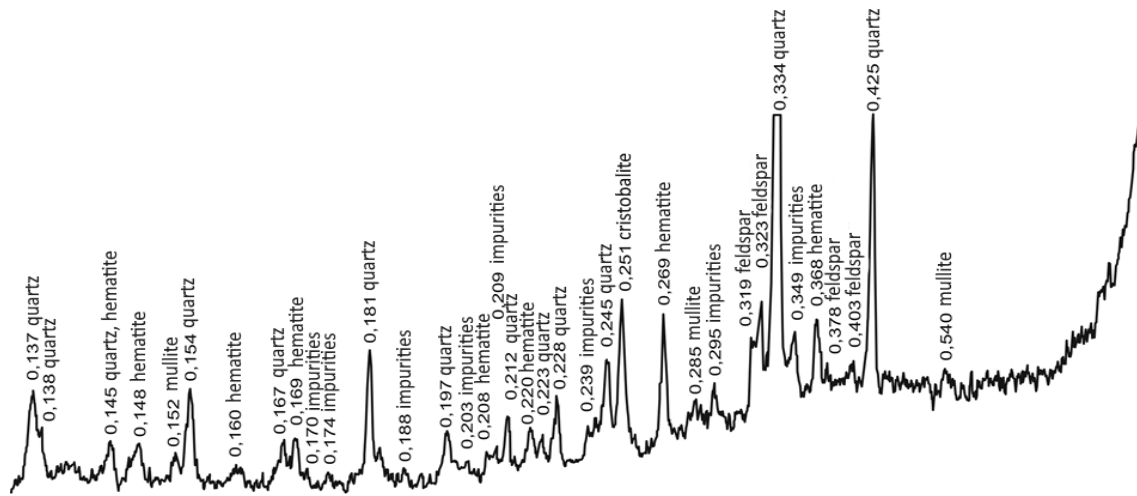


Fig. 4 Diffractogram of coal-mine overburden rocks, argillites, after firing at 1000 °C

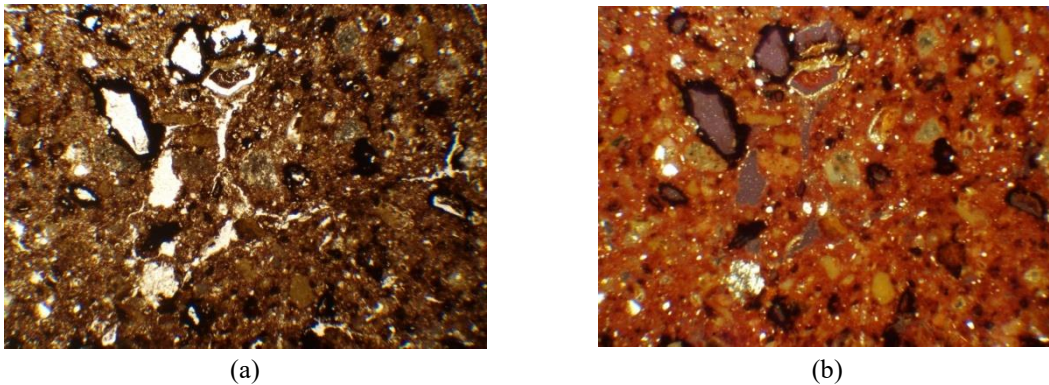


Fig. 5 Microphotographs of the structure of argillites of the +50 m horizon (a); +100 m (b) fired at 1000 °C. Schliph, transmitted light

On the whole, the chemical and mineralogical composition of the overburdened rocks allows concluding that these rocks belong to semiacid raw materials with a low and medium content of coloring oxides. The chemical composition of the mineral part is similar to the chemical composition of clays used for manufacturing ceramic products, which is shown in Fig. 3.

When the samples are fired up to 1000 °C, complete amorphization of clay minerals occurs, 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 has a light brown color, but the core of the dark color remains unburned. This is due to intensive sintering of the marginal zone and in the central zone due to limited oxygen access, whereby the organic matter does not have time to burn out. Reflections of quartz, feldspar, hematite, mullite, and cristobalite are preserved on the diffractograms (Fig. 4).

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 is observed, with an increase in the amount of liquid phase, linking relicts of quartz and ore minerals.

Table 5 Mineralogical composition of ceramic samples from mudstones after firing

Name of raw material	Terrigenous material		Newly formed minerals		
	quartz	feldspar	mullite	cristobalite	haematite
argillite					
horizon +50 m	+	+	+	+	-
horizon +100 m	+	+	+	+	+
horizon +150 m	+	+	+	+	-

Table 6 Physical and mechanical characteristics of samples

Name raw materials	Max. firing temperature, °C	Total shrinkage, %	Compressive strength, MPa	Water absorption, %	Frost resistance bone, cycles
Horizon +100					
argillite	950	4,50	22	12,3	50
	975	4,62	28	11,5	75
	1000	4,80	31	8,6	75
aleurolite	950	3,60	19	12,4	35
	975	3,72	23	11,2	50
	1000	3,81	26	10,8	75

Microphotographs of the structure of argillites of the horizon +50 m, +100 m, burnt at 1000 °C. They are presented in Fig. 4.

In order to establish the peculiarities of mineral formation in ceramic materials from coal waste during firing, we analyzed the intensities of diffraction maxima of the main mineral phases and indirectly estimated the quantitative content of new formations in samples fired at 1000°C (Table 5).

It is revealed that the main structure-forming phase in samples from coal mining waste is mullite, the first crystals of which appear already at 850°C. The intensity of diffraction maxima of mullite is almost twice as high as that of hematite and cristobalite.

The physical and mechanical characteristics of samples from overburdened rocks of coal mining are given in Table 6.

Physical and mechanical tests of the burnt samples allowed us to establish that the highest strength characteristics have samples burnt up to 1000°C. Frost resistance was 35-75 cycles of alternate freezing and thawing without signs of destruction (delamination and peeling). A distinctive feature of the firing mode is the slowing down of the heating rate of products in the interval of organic matter burnout (800-975 ° C). The maximum firing temperature is 1000 °C, duration of isothermal holding - is 2 hours.

On the basis of the conducted research, the rate of temperature rise 100 deg/h, up to 800 °C and 20 deg/h, up to the maximum temperature of 1000 °C is accepted.

4. Conclusions

The results of studying the regularities in the change in the composition and properties of coal mining overburden rocks as raw materials for making ceramic products have allowed the identification of the following facts:

- The physico-mechanical properties of the rocks downgrade with the change in the overburden rock occurrence horizons from +50 m to +150 m, which stems from the reduction in the density rate and more significant weathering;
- In terms of chemical and mineral composition, coal mining overburden rocks are similar to argillous raw materials and belong to semiacid materials with a low content of coloring raw material oxides (Stolboushkin 2016);
- It has been established by diffractometric and petrographical tests that coal mining overburden rocks belong to kaolinitic hydrous mica raw materials with small amounts of quartz, feldspar, ferrous minerals, and organic matter.

Considering the foregoing, it can be concluded that, in terms of physico-mechanical properties, and chemical and mineralogical composition, coal mining overburden rocks are similar to conventional argillous raw materials and can be used after appropriate processing to make ceramic products.

Thus the utilization of the coal mining overburden rocks from the Ekibastuz basin will allow building industry enterprises to expand the range of ceramic building materials produced in the region, cut their cost, and fill the lack of standard argillous raw materials, whereas the coal industry enterprises producing overburden rocks as production waste will make lower environmental protection payments, cut the costs of maintaining the dumps, and improve environmental conditions.

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