

Article

Estimation of Chemical and Mineral Composition, Structural Features, and Pre-Firing Technological Properties of Waste Coal Heaps for Ceramic Production

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Abstract: The relevance of the investigation and creation of a new non-traditional raw material base for wall ceramics for the south of Russia is shown in connection with the decreasing availability of traditional raw materials—loams. Characterizations of the mineral and chemical constituent rock formations of the rocks composing the dumps of coal waste heaps and enrichment plants are given. A serious constraint for the industrial development of coal wastes is the requirement for a great variety of mineral constituents. The chemical and mineralogical compositions and the pre-firing ceramic properties of the waste coal heaps are studied and presented in detail. It is mentioned that fine and thin materials contain coal in an increased amount; due to this, they cannot be considered as the main raw material for the production of wall ceramics. The materials of the medium-sized grain group (2.0–5.0 mm, sifting) can contain up to 2–3% of coal and are most often represented by a mixture of mudstones, siltstones, and sandstones, with the predominance of one or another type of rock. The granulometric composition and the content of large-grained inclusions, molding moisture, plasticity, cohesiveness, desiccation properties, and air shrinkage were studied and determined. It is concluded that the middle group of waste coal heaps in particular are of the greatest interest as a basic raw material for the production of wall ceramic products.



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Keywords: products of processing of waste heaps; wall ceramics; technogenic raw materials; chemical composition; mineralogical composition; pre-firing properties

1. Introduction

The amount of technogenic waste is steadily increasing all over the world. This fact urges humankind to constantly search for effective ways to utilize and recycle waste. It is also necessary to strive to reduce the use of non-renewable resources and reduce the negative impact on the environment. One technogenic waste is the waste of the coal industry. The use of this is currently very small and amounts to about 10% of the total waste volume.

Active processing of coal waste heaps for the purpose of coal extraction in recent years has significantly increased the interest of the building materials industry in the by-products of their processing. This is primarily due to economic factors, as materials with stable properties and acceptable cost can be obtained. In the process of extracting coal from waste heaps, a number of products are formed that differ in material and grain composition. In addition to coal, which is contained in amounts from 10 to 20%, the main rocks composing the waste heaps are mudstones, mudstone-like clays, siltstones, and sandstones.

Currently, coal-mining or coal-processing enterprises have accumulated quite a large amount of technogenic raw materials of coal series, which can be used in the production of building materials [1,2].

Coal dumps, which are formed as a result of coal mining and preparation, are the technogenic deposits of coal series. These include geological remains represented by mine

and coal enrichment plant dumps. All components that are excluded from the final product of a coal mining or coal enrichment enterprise are raw materials that could be used for the production of new products, for example, building materials. Consequently, coal wastes can be classified as mineral resources that are incidentally extracted from the subsoil during the extraction of the main mineral, i.e., as technogenic raw materials; now or in the future, these can be involved in economic turnover, and these are becoming increasingly valuable every year.

Technogenic raw materials include carbon-containing rocks, carbonaceous aggregates, inclusions in coals, coal, and coal–rock slurries. They have the following characteristics:

- The rock mass stored in rock dumps, tailings, and sludge storages leaves about 85% of the volume of rocks extracted from the subsoil during coal mining, i.e., according to the scale of accumulation of such rocks on the earth's surface, dumps represent a phenomenon of a geological scale.
- Under the influence of hypogene and technogenic factors, the rock mass stored on the surface ultimately acquires a new state of aggregation, its quality characteristics are transformed, and new authigenic and technogenic minerals are formed.

The rocks composing the dumps, as shown by domestic and foreign experience, can serve as an important source of raw materials for enterprises of the construction industry and other sectors of the economy [3–6]. A very important limiting factor in the use of coal mining waste is the presence of sulfur in them. For example, its content in the rocks of the central Donbass can reach up to 2%.

The general and special properties of any material are determined by its substantial composition—the chemical and mineral compositions—and its structural features. Knowing these indicators, the kind of pre-firing and firing properties that certain materials will have can be predicted, even without conducting special tests. In our case, as the main base material for the production of wall ceramics, the material of interest is waste coal heaps (WCH). In addition, knowing the composition and structural features of the initial materials, it is possible to plan the approximate composition of the charge, the technology of preparing raw materials, and the molding products. Therefore, study of the chemical and mineral compositions and the structural features of raw materials is the first and essential stage of any technological research undertaking.

2. Materials and Methods

Standard testing methods for ceramic raw materials were used in accordance with Russian GOSTs in order to ensure a comprehensive study of the physical and mechanical properties and structure of waste heap processing products [7–11].

Laboratory technological samples of WCH, weighing 50–100 kg, were selected in specific areas of their accumulation, storage, and processing; they were formed from ordinary point samples that did not contain plant, soil, or foreign inclusions. The basic samples of raw materials were pre-dried to an air-dry state, crushed on a laboratory jaw crusher if necessary, and fractionated according to grain composition.

The chemical composition of the WCH samples was determined in accordance with the requirements [10]. The mineral composition of the WCH was determined by optical microscopy (conventional digital and polarizing microscopes) and X-ray analysis. Electron-microscopic studies of morphology and elemental composition were performed on a Tescan VEGA II LMU scanning electron microscope integrated with the INCA ENERGY 450/XT energy-dispersive microanalysis system. The samples were studied in the form of individual grains. The surface of the samples was sputtered with carbon.

To assess the pre-firing technological properties of waste dump processing products, laboratory tests were carried out using the VNIISTrom method, which is focused on the production technology of ordinary and facing bricks and large-format ceramic stones using plastic molding [10].

After grinding, the raw materials were moistened to the required molding moisture and stored in conditions excluding drying of the mass for 24–48 h. Standard samples were

formed next: cubes with dimensions of $50 \times 50 \times 50 \text{ mm}^3$, bricks with dimensions of $67 \times 30 \times 15 \text{ mm}^3$, and beams with dimensions of $135 \times 30 \times 15 \text{ mm}^3$ and $160 \times 40 \times 40 \text{ mm}^3$. Brick samples of standard sizes were made for control tests: $250 \times 120 \times 65 \text{ mm}^3$ and $200 \times 100 \times 50 \text{ mm}^3$.

Freshly molded samples were kept in natural conditions for a day in the absence of draughts, and then dried in a desiccator for 24 h at a maximum temperature of $90 \pm 5 \text{ }^\circ\text{C}$. After drying, the samples were examined with a focus on all the changes in their appearance; air shrinkage was determined as well.

The determination of sensitivity to drying was carried out according to the method of A.F. Chizhsky [10]. This method is based on irradiating the sample with a powerful radiant heat flux until the first cracks form. The irradiation time, measured in seconds, is an indicator of sensitivity to drying: less than 100 s—highly sensitive; 100–180 s—medium sensitivity; more than 180 s—low sensitivity.

The plasticity of the molding mass was determined according to the method of A.M. Vasiliev [10], based on the determination of the difference in the moisture content of the ceramic mass, which corresponds to the lower yield stress and the rolling limit.

The connectivity was determined by the results of determining the bending strength of samples dried at a temperature of $105 \text{ }^\circ\text{C}$ and molded at normal molding humidity using a plastic method.

The compressive and flexural strength of the samples was determined in accordance with GOST 21216 and GOST 530 [7,10].

Statistical methods of control helped to ensure the reliability of the obtained results during the research. The acquired data obeyed the law of the normal distribution of random errors.

When processing received data, the confidence interval (with a confidence probability of 0.95) and the mean square deviation were determined as well. Repeated experiments and analytical works were carried out to confirm the obtained data.

3. Results

Our study of chemical and mineral compositions of WCH throughout the years, which are closely related, showed that they do not change significantly [12–19].

Table 1 shows the average and limiting chemical composition of WCH compiled from 28 analyses conducted at different times.

Table 1. Limiting and average chemical composition of WCH, % by weight.

WCH	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃ gen.	CaO	MgO	SO ₃ gen.	K ₂ O	Na ₂ O	P ₂ O ₅	TiO ₂	MnO
9–11	52.81– 58.58	16.93– 22.76	4.30– 7.11	1.02– 3.05	1.90– 3.84	0.21– 0.70	3.68– 5.06	0.28– 1.26	0.15– 0.29	0.70– 0.94	0.06– 0.14
9.82	54.82	19.07	5.94	1.52	2.41	0.29	4.21	0.80	0.21	0.81	0.09

In terms of chemical composition, screenings have no fundamental differences from typical clay raw materials and are characterized by an Al₂O₃ content of 16% to 22% and a potassium oxide content of more than 4%.

As can be seen, the chemical composition of WCH varies within relatively small ranges. The SiO₂ content varies from 52.81 to 58.58%. Silica in WCH is a part of clay minerals, micas, and feldspar, and is also represented by quartz. Here, we can say that the more silica is contained in WCH, the more they are sanded, i.e., they contain more quartz grains. The alumina content ranges from 16.93 to 22.76%. Alumina is included in the WCH in the composition of micas, clay minerals, and minerals from the feldspar group. In analogy with clay raw materials, according to the normative-technical documents, WCH can be referred to the group of semi-acidic raw materials; the amount of Al₂O₃ is optimal for the production of many types of ceramic bricks. The content of iron oxides ranges from 4.30 to

7.11%. This is a fairly large amount and, by analogy with clay raw materials, according to the regulatory and technical documentation, WCHs can be classified as a group with a high content of coloring oxides. We can confidently say that a ceramic shard based on WCH will have a dark, red-brown, or brown color depending on the firing temperature. In addition, iron oxides in ceramic masses work as melts. Therefore, ceramic masses based on WCH can be expected to sinter at low temperatures [20–24]. It should also be taken into account that iron oxides change their valence when the gas medium changes; Fe_2O_3 turns into FeO with all the ensuing consequences. Therefore, with such an amount of iron oxides, it is necessary to pay attention to the firing mode.

The amount of alkaline-earth oxides in the composition of WCH is not high. Their presence may indicate the presence of carbonates—calcite and dolomite. Calcium oxide is also a part of basic plagioclases—*anorthite*, *bitovnite*, *labrador*—and, in small amounts, it is a part of some clay minerals. The presence of magnesium oxide in the composition of WCH is also associated with the presence of magnesian chlorites and to a lesser extent may be due to the presence of *serpentine*— $\text{Mg}_6[\text{OH}]_8(\text{Si}_4\text{O}_{10})$ —as a secondary mineral. Such minerals containing magnesium oxide as *montmorillonite*, *vermiculite*, *palygorskite*, and *sepiolite* are practically not found in the composition of WCH.

A fairly large amount of alkaline oxides—potassium and sodium—is contained in WCH. At the same time, potassium oxide contains almost 5 times more than sodium oxide. Such an amount of alkaline oxides implies the fusibility of WCH, since potassium and sodium oxides are strong melts.

The content of sulfur oxides is not high and does not exceed the required values—no more than 0.5–2.0%—depending on the type of products. For ordinary bricks and ceramic blocks, this value is no more than 2.0%. The content of titanium, phosphorus, and manganese oxides does not exceed the average values for clays, sandstones, and siltstones. In such small amounts, these oxides do not have any noticeable effect on the technological properties of WCH or the properties of finished products. In general, the chemical composition of WCH is favorable for the production of ceramic wall materials, and it is almost the same as semi-acid (Al_2O_3 content from 14 to 28%) dark-burning clay raw materials (Fe_2O_3 content more than 3%).

The complex of methods using qualitative and quantitative X-ray phase analysis, as well as methods of petrochemical recalculations allowed to establish that the main minerals composing WCH are hydrous mica: in the form of *chlorite*, *illite*, *hydromuscovite*—25–35%; mica, mainly in the form of *biotite* (≈ 5 –10%); feldspars and plagioclases of different compositions—35–40%; quartz—25–35% (Figure 1).

According to the results of the radiograph analysis, the mineralogical composition of the screenings is represented by several minerals. Hydromica (*illite*) is present in all samples, as indicated by peaks at 4.48, 4.99, and 9.99 Å. As a rule, it is the main mineral. However, judging by the intensity of the peaks, it has a different degree of structural perfection and is contained in different mounts in samples from different deposits. Hydromica (*illite*) is a typical clay mineral belonging to the hydromica group. It has the same structural package of 2:1 type as *montmorillonite*; however, unlike it, the tetrahedral layer always contains aluminum ions isomorphically replacing silicon ions, and the resulting charge of the package is compensated for by potassium ions. The thickness of the packet is approximately 1 nm. In addition to K^+ , small amounts of Mg^{2+} , Ca^{2+} , and H_3O^+ (hydroxonium) may be present in the interpacket space. These cations bind the packets quite firmly and polar water molecules cannot penetrate between them and cause swelling. Hydrous mica particles slowly dissolve in water. Hydromica is usually found in combination with other clay minerals, forming joint, mixed-layer formations. Hydromicas are the most common minerals in shales and mudstones. There is a correlation between the content of hydromica and the content of potassium oxide.

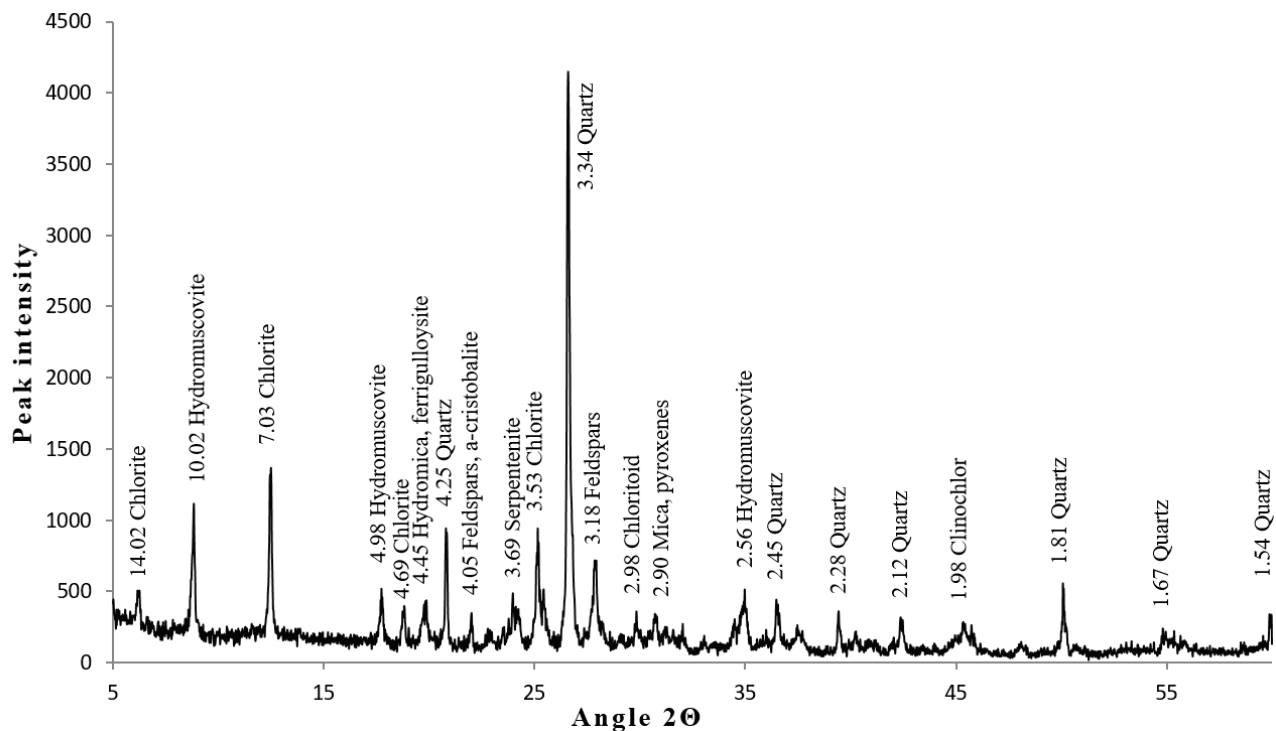


Figure 1. Radiograph WCH of the Eastern Donbass.

All samples contain quartz, which is a terrigenous impurity. Due to its high crystallinity, its diffraction peaks (3.34, 1.54, 1.67, 1.81, 2.12, 2.28, 2.45 Å) have significant intensity. Also, almost all samples contain feldspars and plagioclases (orthoclase, albite, anorthite—2.56, 3.19, 4.24, and 4.71 Å).

Almost all radiographs show peaks of chlorite, clinochlorite, sheridanite—3.53, 4.71, 7.07, 14.2 Å, and others. Chlorite is a mica-like mineral consisting of alternating mica- and brucite-like layers. Due to the nature of the bonds within and between the packets, chlorites usually do not swell in water. They are always found mixed with other clay minerals. The particle size of chlorites is similar to illite clay minerals. Chlorite does not have a constant chemical composition; therefore, different varieties of chlorite are distinguished, having their titles.

It should be said that all the minerals are in very close contact with each other and they are as if bound by silica and ferruginous cementing mass. The water absorption of WCH usually does not exceed 1–2%, porosity—reaching no more than 3–5%. Compressive strength on average varies in the range from 10 to 40 MPa.

As impurities, there are always coal particles in the form of intergrowths with the main rock and silt fraction—up to 3%, iron hydroxides, as well as magnetite, sphene, rutile, garnets, zircon, and other accessory minerals—which do not have a special effect on the technological properties of WCH and properties of finished products. Such a mineral composition is also characteristic of the WCHs of many coal refineries in the Rostov region. This mineral composition determines the low plasticity of the WCH. It is possible to increase the plasticity and improve the formability of WCH by fine grinding and curing them in humid conditions. Radically increasing the plasticity and improving the formability of WCHs are possible by introducing plasticizing additives. However, it is possible to talk about this specifically only after determining the pre-combustion properties. The chemical and chemical-related mineral compositions of WCHs, due to relatively high content of potassium and sodium oxides, are favorable for strong sintering at firing temperatures up to 1100 °C.

Studies have shown that WCH can contain varying amounts of coal component—from 5–10% to 35–40%. Moreover, the use of modern technologies makes it possible to regulate

the coal content during technological processing. Ultimately, it depends on the requirements of individual consumers. Coal is contained in two forms: as individual particles of pure coal and in the form of intergrowths of coal with other rocks—charcoal mudstones (Figure 2). There is no clear boundary between these forms of coal occurrence, but as observations have shown, pure coal particles predominate in the total mass. Studies have shown that there is no difference in coal content depending on the fractional composition—the coal content of the different fractions is about the same.

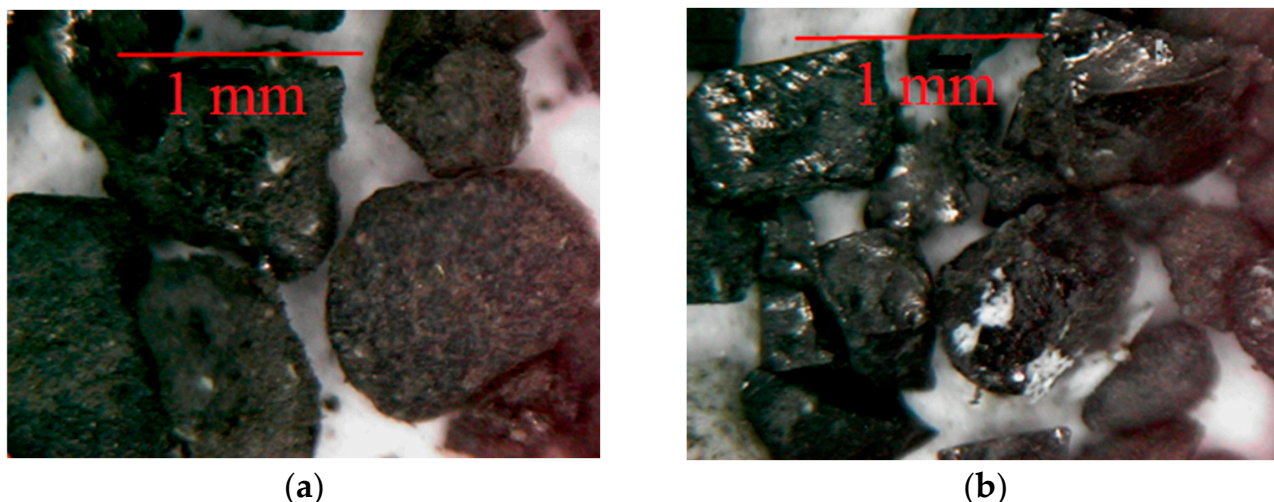


Figure 2. Intergrowths of coal particles with host rocks of fraction 0.63–1.25 (a) and particles of pure coal of fraction 0.315–0.63 mm (b).

Depending on the content of the coal component, we conditionally divided WCH into three groups: those with low content—an amount of coal of up to 10%; those with medium content—an amount of coal of 10–25%; those with high content—an amount of coal of more than 25%. For wall ceramics technology, it can be said that the former can be used as the main raw material with the addition of 10–20% of the plastic component. The second can be included in the raw material mixture from 30 to 80% depending on the specific coal content; the total amount of coal content in the raw material mixture should not exceed 8%. The third category is in fact already low-quality fuel and its content cannot exceed 20–30%. From an economic point of view, all three groups are very attractive, because the cost of a calorie of heat for WCH is 10–20 times lower compared to that of pure coal.

The ash content of coal particles is 5–8%; the yield of volatile components is 3–4%. This is an important argument in determining the optimal firing mode of WCH-based products. The removal of volatile components occurs in the region of 300–350 °C, which is confirmed by the exothermic effect at a temperature of 321 °C (Figure 3). The removal of bound water in clay minerals with a mass loss of about 3–4% occurs in the temperature range of 400–600 °C. Anthracite combustion occurs at 630 °C, which is clearly visible from the exothermic effect and mass loss. However, when one is firing bricks based on WCH in industrial furnaces, this occurs at a temperature of 700–750 °C [25].

Based on the mineralogical and petrographic composition of the WCH, we conditionally divided it into three groups. Group 1 included materials where the main rocks, in addition to coal, were mudstones and mudstone-like clays. The second group included materials where the main rocks were siltstones and sandstones. And the third group included materials where the presence of both rocks was observed.

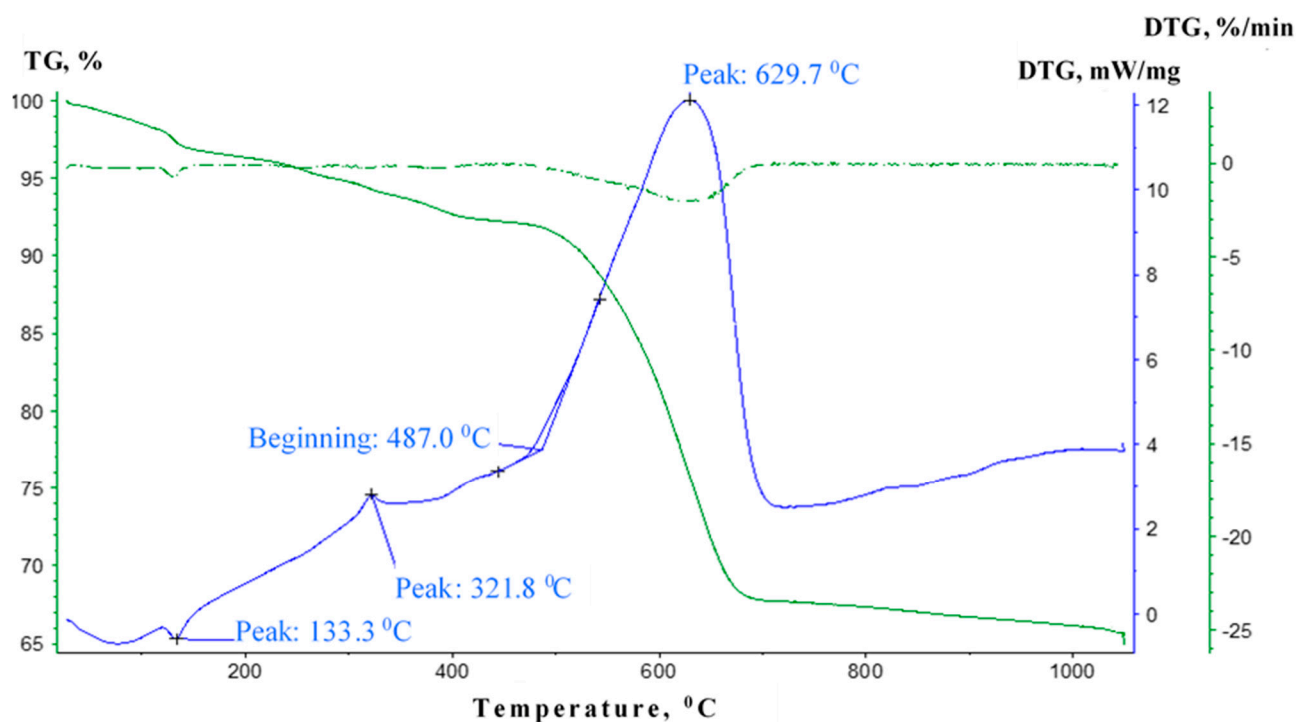


Figure 3. Thermogram of WCH with coal content of about 40%.

The mineralogical composition of the WCH depends on the predominant component. If this is the first group, then the mineralogical composition will be represented by hydromicas, micas, kaolinite, chlorite, quartz, and feldspars. If it is Group 2, then the main minerals will be feldspars and quartz. In the third group, then minerals of both groups are present. There is no clear boundary between these groups, but classification by mineralogical composition is necessary, as this determines the technological features of a particular technogenic raw material. Thus, materials of the first group have a small cohesion and plasticity, which can be increased by additional grinding; accordingly, it is possible to obtain products by extrusion and soft molding. Materials of the third group, in principle, do not have plasticity, and products based on them can only be molded using compression molding or with the addition of a plastic component.

The study of WCH chips under an electron microscope (Figures 4 and 5) confirmed their aleuopelite and aleuropsamite structure and layered texture. Moreover, the change in mineral composition in structure occurs perpendicular to the layering, i.e., each thin layer differs in mineral composition. This is natural, since the formation of these rocks occurred by sedimentation in water basins and the accumulation of certain particles and minerals depended on the speed of the water, the distance from the shore, and the primary material of backwashing from the land. The layered texture determines that, when grinding WCH, the destruction of grains will occur along the weakest interlayers—mica–clay; as a result of this, the crushed particles will have a flattened shape.

Separate recurring thin interlayers are composed of subparallel-oriented microlayers of hydromuscovite (product of partial hydrolysis of muscovite with K_2O content of 7–11%) with the size of tens of microns, immersed in a matrix of more microcrystalline illite with admixture of Fe–Mg chlorite, plagioclases, and aqueous iron oxides. Small silty grains of quartz and feldspars (potassium feldspar, plagioclase) and accessory zircons are present in subordinate amounts.

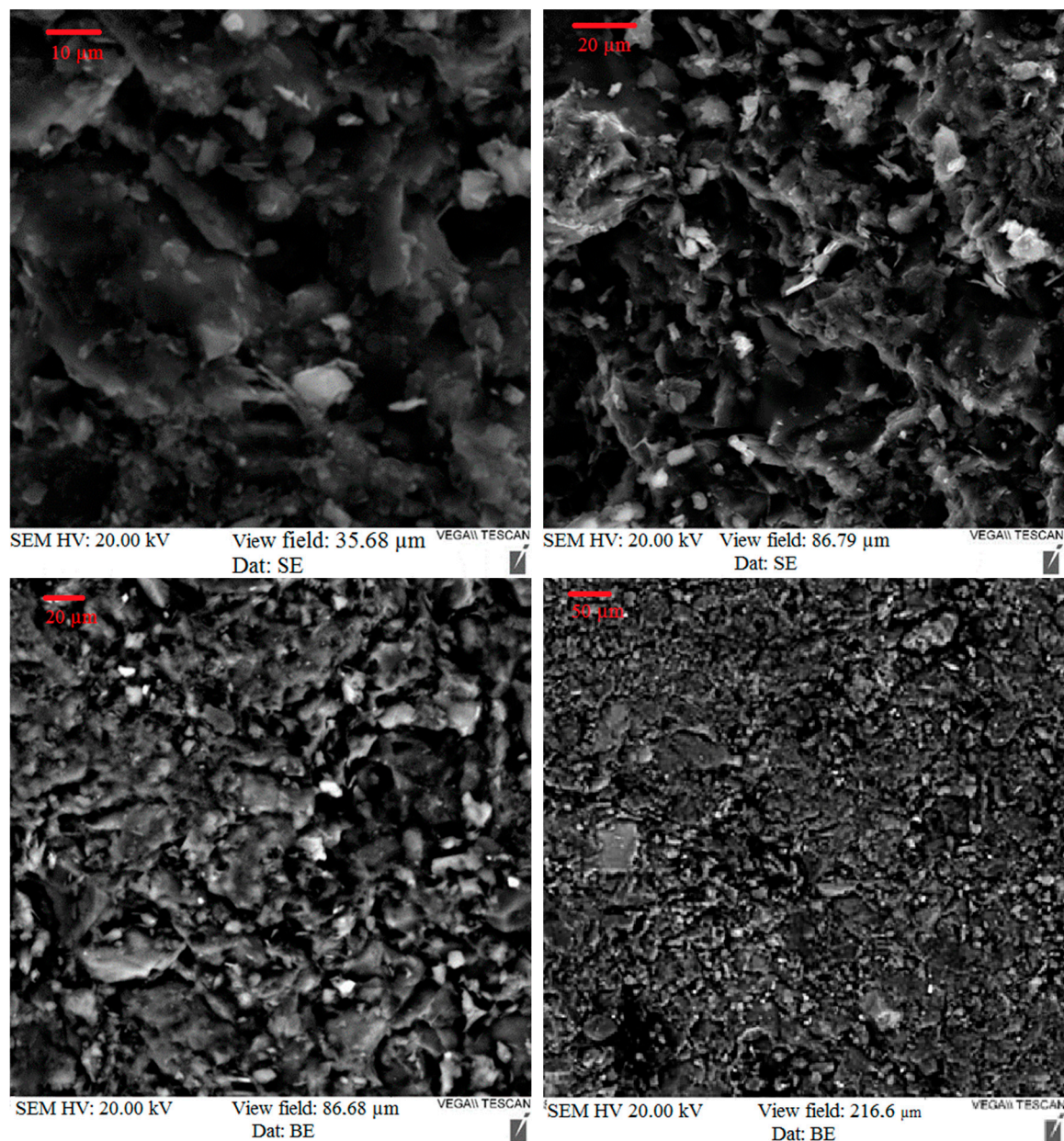


Figure 4. Photos of WCH grain chips under an electron microscope.

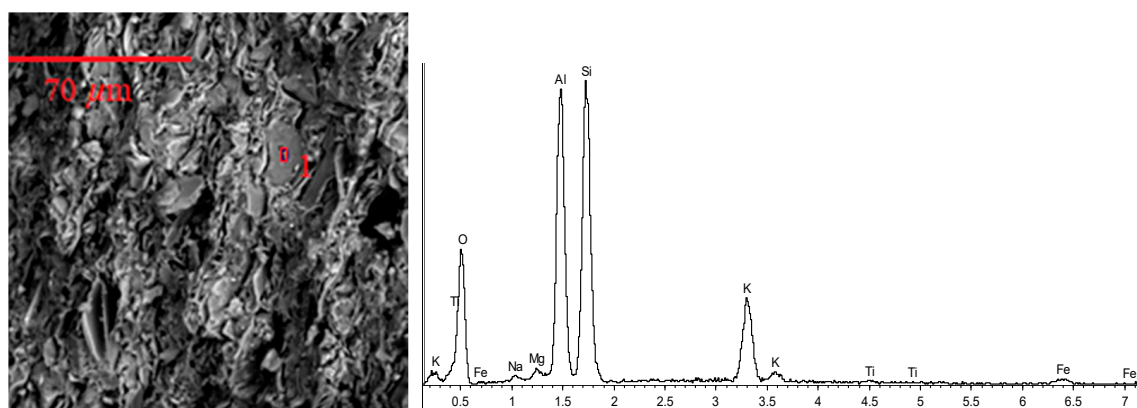


Figure 5. Microphotograph and spectrum of hydrous mica grains.

In general, the chemical and mineral compositions and structural features of WCH predetermine the possibility of their use as the main raw material for the production of ceramic wall products, taking into account the use of additive materials to improve their technological properties and reduce the cost of products.

4. Discussion

It is impossible to design the properties of finished products without a detailed study of the pre-firing technological properties of raw materials. Studies of pre-firing properties consist in determining the particle size distribution and content of coarse-grained inclusions, molding moisture, plasticity, cohesiveness, drying properties, and air shrinkage.

To study the grain composition, we took samples at several large enterprises which are engaged in the processing of the coal dumps of Eastern Donbass.

Table 2 shows the average particle size distribution of WCH. It is characterized by a sufficiently high modulus of fineness; according to this indicator, these can be attributed to the group of sands with increased fineness, with a small amount of a fraction of 0.16–0.63 mm and less than 0.16 mm.

Table 2. Averaged granulometric composition of waste coal heaps.

Residue Title	Residues, % by Weight, on Sieves					Passage through Sieve with Mesh No. 016, % by Weight
	2.5	1.25	0.63	0.315	0.16	
Partial	28.3	31.8	14.7	11.5	7.3	6.4
Complete	28.3	60.1	74.8	86.3	93.6	–

The fineness modulus varies from 1.90 to 2.92 units, which is typical for small, medium, and coarse sand groups. The average value of the fineness modulus is about 2.4 and belongs to the medium group. When comparing the grain composition of the studied material with the normative and technical documents for it, WCH falls into the group of sandy particles—from fine to coarse—where the content of medium-sized particles prevails. WCH grains have a flatter shape, sometimes sharply angular or with slightly rolled edges, which is due to both the characteristics of the constituent rocks and the methods of processing (crushing, sieving).

The use of PP in its original form with a particle size from 2 to 6 mm as a raw material for wall ceramics is impossible; therefore, it is necessary to grind the raw materials to fractions of 0–0.63 mm and 0–0.16 mm.

According to regulatory and technical documentation, particles that do not soak in water, having a size of more than 0.5 mm, are coarse-grained inclusions. They are divided into groups by number, size, and type. Tables 3 and 4 show this classification.

Table 3. By the amounts of coarse-grained inclusions.

Title of the Groups	The Amounts of Inclusions Larger than 0.5 mm, %
With a low content	Less than 1
With an average content	From 1 to 5
With a high content	More than 5

Table 4. By the size of coarse-grained inclusions.

Title of the Groups	The Size of the Predominant Inclusions (Over 50%), mm
With fine inclusions	Less than 1
With medium inclusions	From 1 to 5
With large inclusions	More than 5

Based on the normative and technical documentation [10], the determination of the presence of coarse-grained inclusions is carried out by soaking the raw material and then washing it on a sieve with mesh No. 05, and then scattering the resulting residue on a set of sieves with cell sizes: No. 05, 1, 2, 3, and 5.

Studies have shown that WCHs belong to the group with a high content of coarse-grained inclusions and large inclusions.

In the practice of producing wall ceramics, the concept of relative humidity is often used. Normal molding moisture content for clay raw materials is 18 to 27%. Tests have shown that the normal molding moisture content for WCH is from 18 to 21%.

The main criterion for determining the plasticity of raw materials is compliance with the requirements [11]. The groups of raw materials, arranged by plasticity, are shown in Table 5.

Table 5. Groups of clay raw materials by plasticity.

Group Title	Plasticity Number
Highly plastic	More than 25
Medium plasticity	From 15 to 25
Moderately plastic	From 7 to 15
Low plasticity	From 3 to 7
Non-plastic	Do not give a plastic dough

Before starting the test to determine the plasticity of the mass, we prepared the raw material by grinding and sieving it through a sieve with mesh No. 05. However, even when grinding WCH to a fraction of 0–0.16 mm, the plasticity did not exceed 5–6 units, which corresponds to the low-plasticity group.

Drying of products is characterized by parameters such as air shrinkage and drying sensitivity. Our tests have shown that WCH is a raw material that has low sensitivity to drying.

As extensive practical experience shows, to obtain defect-free ceramic products, air shrinkage should not be more than 7–8% [26–31]. The products of waste heap processing are predominantly insensitive to drying, and, accordingly, have low air shrinkage—no more than 7%. When producing samples, we ground the raw material to a fraction of 0–0.16 mm. When determining air shrinkage, the dried parallelepiped samples had no external defects, cracks, or cuts.

WCHs in their pure form have a low binding capacity, as the bending strength of molded and dried samples is no more than 1–2 MPa. Thus, they can be classified as a group with very low and low mechanical strength. However, when firing these samples, they show a sufficiently high strength. At the same time, the strength of the fired samples strongly depends on the initial raw material degree of grinding and the firing temperature [32–36]. Groups by mechanical bending strength in dry state are given in Table 6.

Table 6. Bending strength groups in dry state.

Group Title	Bending Strength in Dry State, MPa
With very low mechanical strength	Less than 1
With low mechanical strength	1–2
With moderate mechanical strength	2–5
With medium mechanical strength	5–10
With high mechanical strength	More than 10

5. Conclusions

The conducted studies of the chemical and mineral composition, structural features and pre-combustion properties of coal waste heaps allowed us to conclude that they can be used as the main raw material in the production of ceramic wall products. Final

conclusions about the quality and suitability of WHP as a raw material for the production of wall ceramics can be made only after a comprehensive study of their firing properties. It is also necessary to establish the dependence of technological factors on the properties of finished products. Taking into account the above-mentioned data, as well as economic factors—primarily, the minimum cost of raw materials and the minimization of firing costs due to the contained coal—WCHs are promising raw materials for obtaining wall ceramic products with a low cost; this finding applies not only in the south of Russia but also in other coal-mining regions. However, the realization of this plan necessitates the resolution of a number of scientific and practical problems that are connected with the development of testing methods for the given technogenic raw materials and with the selection of the compositions of raw masses. Potential future directions of research in practical terms will be the development of production technology and the selection of optimal equipment.

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