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Coal Mining Waste as Raw Material for the Construction Industry

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Abstract

The resource value of unburned and burnt coal mining rocks was substantiated. The burnt dump rocks of the Olkhovatskaya mine of Donetsk region (Ukraine) contain phases: illite, quartz, brushite, hematite, and albite. Illite and quartz are in all fractions of the rock. The unburned waste pile rocks of Sverdlov and Khmelnitskaya mines of the Lugansk region contain amorphous phases. In the crystalline part of the rocks, the following minerals were found: quartz, hematite, gypsum, clinochlore, and muscovite. The row of descending minerals mass content is as follows: muscovite > quartz > clinochlore. These minerals are allowed in the composition of clay rocks used in the production of Portland cement clinker. When being calcinated, the waste pile rock of the Khmelnitskaya mine, clinochlorine and muscovite decompose to form hematite. Derivatographic analysis confirmed the initial transformation of rock minerals in the waste pile and the ability of the carbonaceous part of the rock to burn. The presence of highly active modifications of silica, alumina, and ferruginous oxides, high adsorption, and hydraulic activity of the burnt rock defines the possibility of its use as active pozzolanic-clay additives to cement clinker and asphalt binder microfiller.

Keywords: *Coal mining waste; Minerals; X-ray phase analysis; Derivatographic analysis.*

1. Introduction

In the modern industry, the concept of sustainable development is expressively relevant. Mankind uses a great number of natural resources for metal smelting, production of binding materials, etc. The depletion of natural resources is one of the problems to be solved effectively. In the construction industry, there is a new trend of replacing primary materials with alternative raw materials. With such an approach, the negative impact on the environment will be reduced, as well as energy consumption, polluting emissions, and the amount of stored waste. The authors of [1] offer to refuse to store the waste, which is to be recycled but to use it as a raw material for recirculation in accordance with the trend of the circular economy. Today, producing fuel and energy from both renewable sources and various types of waste are prospective directions for development [2]. The mining and coal industries produce a large amount of waste in the form of enclosing and overburden rocks that form the dumps [3-4]. This situation calls for the optimization of territorial loads, as agricultural and forestry lands are seized and disturbed, and the expansion of volumes of the associated rocks recirculation and recycling them into the materials with excellent technical characteristics and consumer appeal [5].

Coal mining waste consists mainly of minerals and 20–30 % of organic matter. The material classified as “mineral substance” includes crystalline minerals, as well as inorganic elements in the non-crystalline form. The detrital contribution, biogenic activity, and the formation of authigenic sediments at different stages of the history of coal sedimentation account for the formation of mineral matter [6]. Dumps are influenced by weather conditions and weathering, which begins immediately after sedimentation. Oxidation of organic matter can cause self-heating and spontaneous combustion, resulting in the transformation of organic and mineral substances. The degree of change depends on the rank of the organic matter, the duration, speed, the final temperature of heating, and the degree of air access [7-8]. In [6], the methods used to estimate the content of the mineral substance in coal are considered, as well as the behavior of various mineral substances at high temperatures and the methods of mineralogical analysis of ash.

It is suggested that the overburden rocks of coal mining should be used in the construction of temporary quarries and technological roads. The effective granulometric composition of aggregate with a high static modulus of elasticity and shear resistance is substantiated [9]. In [10] the prospects for using the man-made deposits of the mine dumps in Eastern Donbas are given: waste dumps, coal enrichment wastes in the form of argillite-like clays, argillites, slates, siltstones in the manufacture of wall ceramics. The authors of [11–15] substantiated the use of various wastes of mining and coal industries in brick production, which is considered as an environmentally friendly method of waste management. In some cases, this even implies an improved property of bricks. Bricks with very good physical and mechanical properties were obtained from argillites. For the production of hydraulic lime, the coal washing wastes [16] with the inclusion of coal and clay mineral residues and dolomite sifting in 1:1–1:2 ratios were used. In [17-18], the efficiency of using coal mining waste as a raw material component in cement technology was theoretically proved. Some inert wastes of coal mining and their processing can be used as alternative sources of future environmentally efficient pozzolanic cements [19]. A comprehensive study of the properties of various coal mining wastes will help develop recommendations on their practical use in technologies of producing binding materials.

It is necessary to study the chemical composition and technically useful properties of industrial waste in order to determine the area of its use. We have suggested a method to determine the resource value of industrial waste aiming at their reclamation as technical materials [20]. The method optimizes consistency, increases the efficiency and completeness of the research. The choice of the methods for this research is based on the relevancy of studying the mineral, elemental, and radionuclide compositions of industrial wastes, the structure of their surface, sorption and hydraulic activity, the behavior of minerals when heated. The following methods are suggested for the research – an X-ray gamma-spectrometry, electron-probe microanalysis, a chemical method, petrographic, titrimetric, spectrophotometric, and derivatographic analysis. The composition of the waste can vary depending on the particle size fraction. It is shown [21] that the chemical composition of coke varies depending on the particle size and is determined by the petrographic composition of the coal batch. Therefore, it is necessary to study not only the average waste samples, but also their fractions.

The purpose of the research is to substantiate the resource value of coal mining rocks. In this paper, the dump coal mining unburned rocks of the Khmelnitskaya mine and the Sverdlov mine of the Lugansk region, as well as the burnt rocks of the Olkhovatskaya mine in the Donetsk region have been studied. The research objectives are determining the mineral composition of coal mining waste in order to identify the presence of amorphous structures; studying the behavior of raw coal mining minerals during heating; identifying the areas of practical use of waste piles in manufacturing building materials.

2. Research methodology

The dispersion of waste samples into granulometric fractions was performed using a set of grading screens. The following fractions were identified, mm: > 20, 10–20, 5–10, 2.5–5, 1.25–2.5, 0.63–1.25, <0.63. The particle size composition of the burnt rock of the Olkhovatskaya mine is shown in Fig. 1. The large contribution of the coarse fraction indicates that the original waste rocks were strong and hard, so the material did not undergo natural grinding as a result of roasting, slipping on the slope of the heap of rocks or compression by the overlying layers.

An X-ray phase analysis, which is instrumental in determining the mineral composition of the crystalline part of the samples [22], is performed on a Siemens D500 powder diffractometer in copper radiation with a graphite monochromator. The full-profile diffractograms were measured in the angle range of $5° < 2\theta < (110–120°)$ with a step of 0.02° and an accumulation time of 15 s. The primary phase search was carried out using the PDF-1 card file [23], after which the X-ray diffraction was calculated by the Rietveld method using the FullProf program [24].

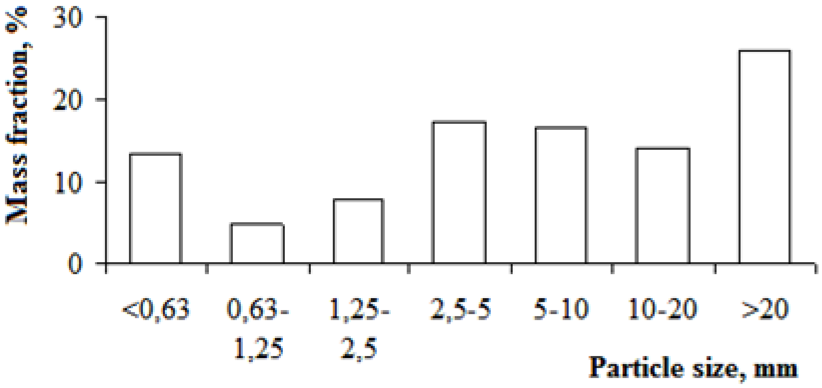

Figure 1. Granulometric composition of the burnt rock of the Olkhovatskaya mine heap of rocks

The derivatographic analysis of unburned dump rocks of coal mining makes it possible to study the transformation of a substance at heating. The analysis was performed on an "MOM 1500" derivatograph with a 10ºC/min—heating rate of a sample weighing 449 mg to a final temperature of 1000ºC. Isothermal curing time was 60 min at 1000ºC in the air environment.

The sorption properties of coal mining rocks were determined under static conditions by changing the concentrations of the absorbed substance (sorbate) in the solution. Methylene blue (MB) was the sorbate; the solution of the initial concentration was 0.1 g/dm^3. The optical density of the solution was determined by the spectrophotometric method with a SPEKOL 11 instrument at λ = 620 nm relative to distilled water.

The hydraulic activity of the rocks was estimated by the amount of absorbed lime CaO. A crushed lot of coal mining rock (10 g) was kept in a CaO solution with an initial concentration of 5.63 % and a density of 0.995 g/cm^3. In the process of interaction with the rock, the concentration of CaO was controlled in time using the titrimetric method.

3. Results and discussion

3.1. Mineral composition of coal mining rocks

The burnt rocks of the Olkhovatskaya mine contain 5 phases (Table 1). It can be assumed that there are no brushite or illite in their pure form in the waste rock; they are formed as a result of the interaction of the rock combustion products in the waste pile with precipitation.

Table 1. The results of X-ray phase analysis of burnt rocks of Olkhovatskaya mine

Phase	Fraction of burnt rock, mm					
	< 0.63		2.5-5		> 20	
	mass content, %	particles average size, nm	mass content, %	particles average size, nm	mass content, %	particles average size, nm
Quartz SiO_2	33.7	97	39.5	86	46.5	100
Brushite $CaHPO_4 \cdot 2H_2O$	10.3	> 500	0.55	> 500	4.36	> 500
Hematite Fe_2O_3	7.79	66	6.50	105	2.96	114
Albite $NaAlSi_3O_8$	8.8	58	2.3	20	–	–
Illite $KAl_4Si_2O_9(OH)_3$	39.4	25	51.1	25	46.1	25

The main mineral components of the studied samples are quartz and potassium hydroxoaluminosilicate (illite), whose share is larger in coarse fractions than in fine fractions. Minor amounts of calcium hydrophosphate (brushite) and sodium aluminosilicate (albite) are contained mainly in fine fractions, and the latter phase is absent in the large fraction.

A positive feature in assessing burnt rocks in terms of their use as aggregates of concrete and mortars is the absence of unburned coal and sulphides (pyrite and marcasite). These admixtures are characterized by high water absorption and capillary suction, hydrophilic nature of the surface and the ability to react with oxygen and water, which causes the changes in the volume of the solidified product.

It is possible to approximately determine the burning temperature of the burnt rock by the occurrence of certain phases. The absence of metakaolinite $Al_2O_3{\cdot}2SiO_2$ and sillimanite $Al_2O_3{\cdot}SiO_2$ indicates that the burning temperature reached ≈ 600ºC. The amorphous oxides are practically non-existent at such temperatures.

The unburned rocks of the waste piles of the Sverdlov and Khmelnitskaya mines. In [25] it was shown that as a significant amount of coal is present in the mine pile rocks of the Ya. M. Sverdlov mine, a wavy background, can be seen on radiographs of dump rocks. The distinct wavy background on the radiograph of the fraction >20 mm of the mine pile rock (Fig. 2) is associated with the presence of the amorphous phase.

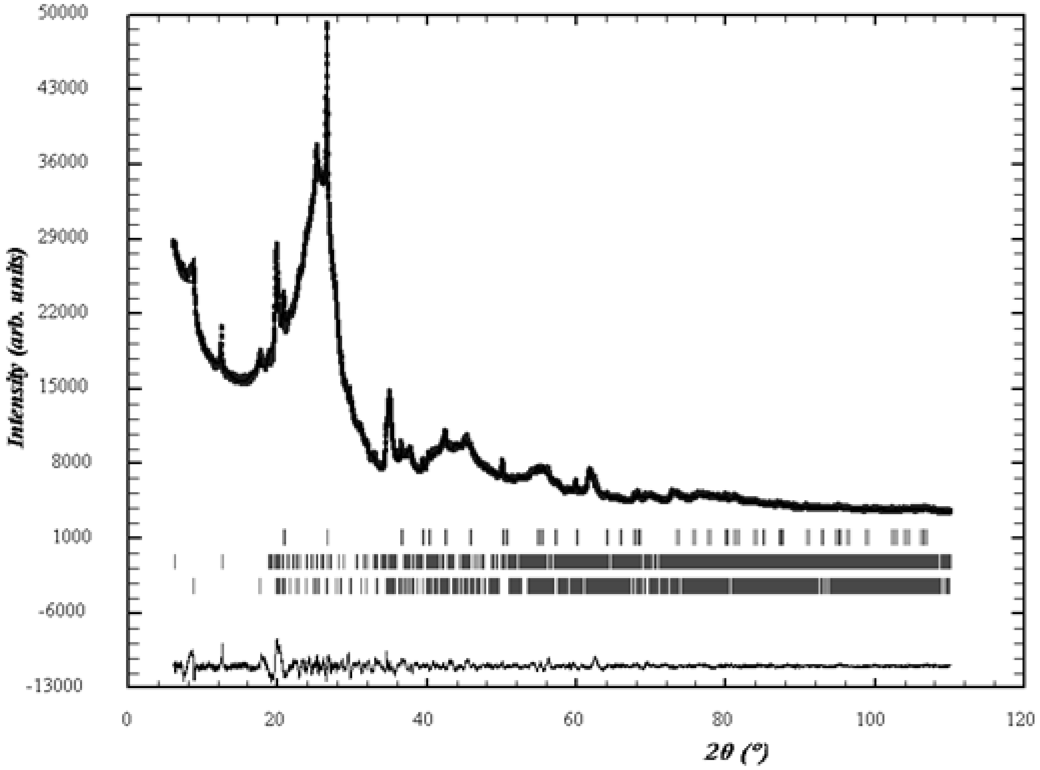

Fig. 2. Diffractogram of a > 20 mm fraction of Sverdlov mine heap of rocks

Several minerals were found in the crystalline part of the dump rocks; their mass content and the average crystallite size are given in Table. 2. Muscovite prevails by mass, then comes quartz, and the least is clinochlore. Clinochlore and muscovite could have formed in the process of chilling hot waste pile rocks by atmospheric precipitation. The presence of these minerals is allowed in the composition of the clay rocks used in the production of Portland cement clinker.

The structures of clinochlorine and muscovite are laminated sandwich-type. Each "sandwich" consists of two flat layers of silicate tetrahedral with a layer of magnesium or aluminum octahedral between them. Such compounds are thermally unstable and, when heated, they split off the water, which is confirmed by the destruction of both phases during calcination of the heap of rocks of the Khmelnitskaya mine (Table 2). Hematite is found as a decomposition product of clinochlorine and muscovite. If during thermolysis, silicates or aluminates of potassium and/or sodium are formed, they can be amorphous.

Table 2. The results of X-ray phase analysis of the samples of unburned dump rocks

Phases	Khmelnitskaya mine rock				Waste heap of rocks of Sverdlov mine			
	Sample 1 waste slagheap rock		Sample 2 calcinated rock		Sample 3 < 5 mm fraction		Sample 4 > 20 mm fraction	
	mass content, %	size, nm	mass content, %	size, nm	mass content, %	size, m	mass content, %	size, nm
Quartz	44.1	> 200	73.5	140	40.3	140	7.7	120
Clinochlore $(Mg,Fe)_6(Si,Al)_4O_{10}(OH)_8$ (chlorite)	10.2	38	–	–	11.1	47	17.4	48
Muscovite $K_{0.94}Na_{0.06}Al_{1.83}Fe_{0.17}Mg_{0.03}(Al_{0.91}Si_{3.09}O_{10})(OH)_{1.65}O_{0.12}F_{0.23}$ (mica)	45.7	32	21.2	35	48.0	45	74.9	25
Hematite	–	–	5.3	–	–	–	–	–
Gypsum $CaSO_4{\cdot}2H_2O$	–	–	–	–	0.7	–	–	–

If the rock stays long in the waste pile, it can be the reason for the transformations of its minerals. The process of weathering of layered minerals involves their transition to clay minerals. Muscovite eventually turns into illites (hydromica), zeolites, and kaolin. One of the causes of weathering is leakage of groundwater. When mica contacts with acidic solutions, potassium is replaced by H_3O^+ ion. Rainwater can be the second reason for weathering; it always has a weak acid reaction (pH = 5.7) when CO_2 is dissolved. This process is very likely to occur in the waste slag heaps at acid rains, which is so characteristic of the industrial Lugansk region. Since the illites are marked by a higher water content than conventional micas and emit constitutional water more easily, then when the rock is heated, the appropriate endothermic effects should be observed. The derivatographic analysis was made to clarify this circumstance.

The results of the derivatographic analysis are given for the dump rock of the Khmelnitskaya mine. On the thermal effect curve – *dTa* (Fig. 3), the peaks which are characteristic of the detected minerals are noted. For clinochlorine at a temperature of 475 ºC, a clear endothermic effect is observed, corresponding to the release of all constitutional water [26]. The intense endothermic reaction at 550–650ºC [26], characteristic of clinochlorine, is somewhat erased, as is the exothermic effect at 820ºC [26], resulting from the interaction of active amorphous SiO_2 and MgO oxides in the solid-state with the formation of magnesium orthosilicate. According to the literature data, two endothermic effects should be observed on the heating curves of muscovite: at temperatures 860°C and 1200°C [26]. The first effect, due to the release of structural water, is noted on the thermogram at 897ºC. The second effect, associated with the destruction of the crystalline lattice, was not fixed in the experiment, since the heating was up to 1000ºC.

The thermal behavior of clinochlore and muscovite largely depends on the degree of the samples grinding. The presence of a clear endoeffect at 185ºC (Fig. 3) indicates a high granularity of the samples, including muscovite [26].

Low-temperature endothermic effects of the heat effect curve (Fig. 3) can belong only to the minerals that bind water loosely. For example, illite dehydration has several stages; therefore, several endothermic effects were recorded on the heat effects curve in the temperature range of 100–400ºC.

In the temperature range from 185 to 510ºC (Fig. 3), endothermic effects associated with several processes are observed. First, volatile substances are released. This corresponds to a wide endothermic minimum, ending at 400ºC. Secondly, the water of the illites, represented by OH^- ions, is removed. The loss of OH^--ions causes insignificant disturbances in the crystalline structure of the illites, which persists to the temperatures of about 750°C [26]. Two small endothermic peaks at 928 and 967ºC can be attributed to the destruction of the residual structure of the illite crystalline grid (850–950ºC) with the appearance of spinel in this temperature range [26]. The exothermic effect at 670ºC is associated with the combustion of carbonaceous matter.

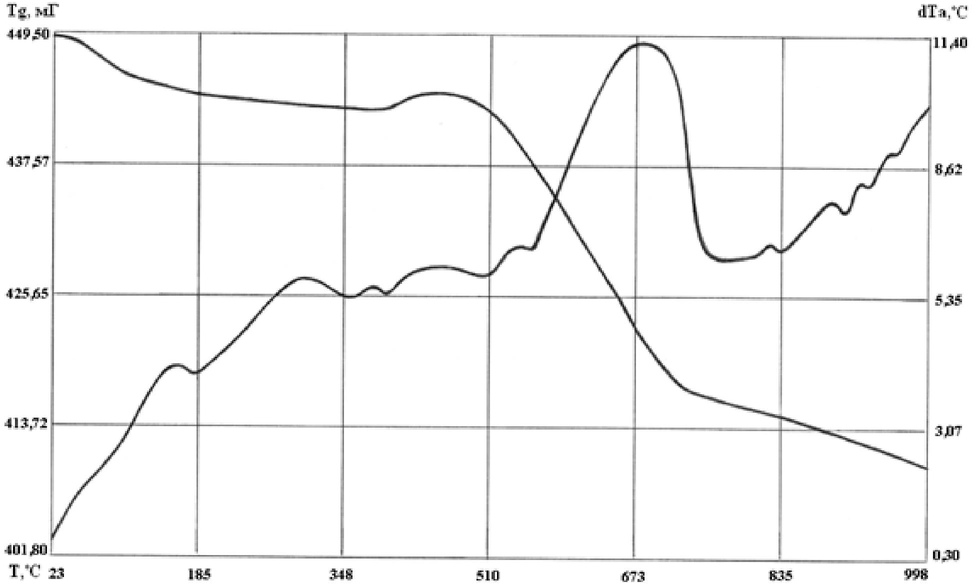

Fig. 3. The thermogram of the dump rock of Khmelnitsky mine: *Tg* – mass loss; *dTa* – heat effects

The mass loss curve – *Tg* (Fig. 3) reflects these processes. During dehydration, a gradual decrease in the mass of the sample up to a temperature of 500°C is recorded as well as a subsequent sharp drop in mass to the temperature of 725°C (combustion process). At higher temperatures, the decrease in sample mass slows down with increasing temperature. Thus, as a result of derivatographic analysis, the initial transformation of the minerals of the waste piles rock was confirmed, and it was shown that the carbonaceous part of the rock could be burned.

The substantiation of the resource value of waste piles is made on the example of the burned waste rocks of the Olkhovatskaya mine. An important indicator is the oxide composition, calculated according to X-ray phase analysis (Table 1). The oxide composition of the waste burned rock fractions, and the modular classification are presented in Table 3.

Table 3. Content of oxides and value of modules for the waste burned rock fractions of Olkhovatskaya mine

Oxide	Mass content of oxides (%) in waste burned rock fractions (mm)		
	<0.63	2.5–5	>20
SiO_2	51.6	56.48	60.4
Al_2O_3	21.92	26.65	23.63
Fe_2O_3	7.79	6.5	2.96
K_2O	4.65	6.03	5.44
Na_2O	1.04	0.27	–
Module	Value of modules for the waste burned rock fractions		
$M_a = Al_2O_3/SiO_2$	0.42	0.47	0.39
M_s $SiO_2/A1_2O_3$	1.73	1.70	2.27
$M_{c.i.}=\frac{Al_2O_3+Fe_2O_3}{SiO_2}$	0.58	0.59	0.44

A comparison of the oxide composition of burned rocks with the data for the coal rocks of the Donetsk coal basin [27-28] showed that the SiO_2 content is less than the lower interval value

(53.1–73.5 %) for the fraction <0.63 mm. The content of Al_2O_3 is at a fairly high level; the interval, according to literature data, is 14.22–27.91 % [10, 27]. CaO content should be between 0.27–3.21 %, but there is no lime in the crystalline part of the fractions studied. The dispersion of literature values for Fe_2O_3 is 5.55–12.18 %; therefore, fractions <063 mm and 2.5–5 mm are in this interval.

The characteristics of burned rocks by chemical composition are not comprehensive for the estimation of their quality and resource properties. The chemical and hydraulic activities of burned rocks, which can be characterized by a system of modules, are very important indicators. The value of the silicate modulus M_s is within the limit of up to 2.4 for acidic, latently active rocks [28]. Using the clay-iron module $M_{c.i.}$ is advisable since the burned rock is ferruginous. The value of $M_{c.i.}$ testifies that the burned rock fractions are highly active ($M_{c.i.}$>0.45), which is associated with the presence of sodium aluminosilicates (albite) and potassium (illite). The values of the modules vary in fractions of the burnt rock. The maximum value of the modulus of activity M_a is for a 2.5–5 mm fraction, the maximum value of $M_{c.i.}$ is for a fraction >20 mm.

The presence of highly active modifications of silica, alumina, and ferruginous oxides in the burning rock confirms the possibility of using it as a pozzolanic-clay component of building materials. When hardening, active forms of oxides form cementing compounds in reactions with lime. This is confirmed by the results of the determination of hydraulic activity when absorbing CaO. Hydraulic activity is equal to mg/g: per 1 day – 211.4, per 3 days – 323.4. Comparison of the obtained experimental data with literature data shows that the amount of CaO absorption exceeds those values, mg/g: for burnt clay – 30 (for 30 days), burnt waste rocks – 50, silica, ferruginous burnet Kuzbass rocks – 40–130 [28]. The absorptive capacity of the studied waste rocks is comparable with quantitative indicators for acidic hydraulic additives: opaline rocks (trippel, diatomites, flasks) – 250–400 mg/g [27-28]. High absorption capacity indicates the possibility of using coal mining wastes as active additives to cement clinker, which should absorb at least 50 mg/g of lime in 30 days [29].

The high hydraulic activity of burnt rocks was confirmed in experiments on determining adsorption activity when absorbing MB in time. The sorption capacity of the rock is equal to mg/g: per 1 hour – 0.77, per 3 days – 0.98. The efficiency of sorption cleaning of the solution for 3 days reaches 99.3 %. Waste burned rocks of the Olkhovatskaya mine can be ranged in a group of adsorbents characterized by very high adsorption activity [28], which, given the absence of carbonaceous impurities during burning, can be explained by the presence of clay particles in a highly dispersed state. Thus, the high values of the hydraulic and adsorption activity of burnt rocks make possible to consider them as an active mineral additive to cement and an active mineral adsorbent – a microfiller for asphalt binders.

Prospects for research are to justify: the use of burnt waste rocks in the production of complex binders when adding them to the clinker as a hydraulically active component during grinding; the use of non-burnt waste piles rocks in the composition of the raw mix for manufacturing aluminous and Portland cement clinker instead of the clay component.

4. Conclusion

It has been shown that the study of the properties and modification of dump rocks under various conditions calls for an integrated approach, including X-ray phase analysis and derivatographic analysis.

The X-ray phase analysis chosen for the research allowed us to identify the minerals of the coal mining dumps that are in the crystalline state, to determine the structure of the crystals, to confirm the presence of the amorphous state of the substances. It was revealed that the mineral composition of granulometric fractions of coal mining rocks is varied. It was also proved that dump rocks have minerals in their composition that are technically valuable in the production of binding materials.

Thermal analysis has shown the transformation of the minerals in dump unburned rocks during heating and the ability of the carbonaceous part of the rock to burn.

The presence of highly active modifications of silica, alumina, and ferruginous oxides, high adsorption, and hydraulic activity of the burnt rock defines the possibility of its use as active pozzolanic-clay additives to cement clinker and an active mineral adsorbent – asphalt binder microfiller.

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