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USE OF ROCK REFUSE OF COAL EXTRACTION IN MANUFACTURE OF BUILDING MATERIALS

The work's aim is the finding out possibilities of use of «Khmelnitskiy» and «Ya. Sverdlov» mines rock refuse (Lugansk Region, Ukraine) in manufacture of building materials. The research methods of X-ray diffraction, petrographic, spectrophotometric, titrimetric analysis and electron probe microanalysis were used to study the composition and properties of rock refuse.

The mineralogical and oxide compositions of rock refuse of coal extraction and its clinkered samples were researched. The following minerals are present in the crystal part of rock refuse: quartz, clinochlore (chlorite), muscovite. Clinochlore and muscovite refer to clay minerals which can be used in the manufacture of Portland cement clinker.

The high hydraulicity and adsorption activity of rock refuse were determined. The adsorptive property of rock refuse is comparable with indicators for acid hydraulic additives and diatomite – 250-400 mg/g. High adsorbing ability is evidence of considerable hydraulicity. High hydraulicity indirectly proves to be true by determination of adsorptive activity of rock refuse. The efficiency value of solution sorption clearing proves the high sorption capacity of rock refuse: during 15 mines it reaches 67.5 %; within 3 days it reaches the maximum value of 99 %.

High concentration of glass phase, aluminate and iron(III) oxides, high values of alumina and clay-ferriferous modules, high hydraulic and adsorptive properties of rock refuses make possible their utilization in manufacture of aluminous cement as correcting additive or active additive to Portland clinker or in manufacture of calcic-slag binders. It was shown that the recycling of rock refuse will allow providing the building industry with readily available and cheap mineral raw materials and improving ecological conditions in industrial regions.

Key words: *rock refuse, coal extraction, building materials, binding materials*

I. Introduction. The majority of chemical elements are encountered in the nature in connected condition together with by-products that cause economic feasibility of complex use of raw materials for manufacturing of qualitative production. With a rise in prices for raw materials processing, waste and used materials should be applied in the large. The operating ratio of natural resources thus increases both technical and economic indicators of manufacture improve. Solid waste of mineral origin of the mining and coal industry is collected in a considerable quantity. One of perspective directions of their use is manufacture of building materials. Use of production wastes will allow obtaining cheap binding substances.

One of the varieties of solid waste of coal output are dead rocks accompanying coal layers. Their practical use in the building industry is limited for several reasons. First, exploitation of waste heaps remains a technically difficult and unsafe problem. Secondly, rock refuse of waste heaps is non-uniform according to the structure and roasting degree, therefore in each case preliminary mineralogical research [1, 2] is necessary. At present, rock refuse of coal extraction is not fully studied. Accumulation of data bank of petrographic and mineralogical information can promote the improvement of quality and perfection of the production technology of binding materials and creation of new ones.

Rock refuse of coal extraction can be used in manufacture of binding materials as replacement of raw mix components of portland-slag cement manufacture, as a Portland-slag cement component at joint crushing of cement clinker and waste, as correcting and active additives, etc. To determine the way of rock refuse application it is necessary to determine its oxide, mineralogical structure, to characterize hydraulic properties and to investigate the behaviour of minerals at high-temperature burning.

II. Formulation of the problem. The objective of the given work was studying of mineralogical characteristics and hydraulicity of rock refuse of «Khmelnitskiy» and «Ya. Sverdlov» mines of Lugansk Region, Ukraine and finding out possibilities of their use in manufacture of building materials.

Element compositions of rock refuse are determined by means of the method of electron-probe microanalysis INCA applying JSM-820 scanning electronic microscope. Oxide structures have been calculated according to element compositions.

The mineralogical composition of rock refuse is determined with the help of the roentgen-phase analysis with application of Siemens D500 powder diffractometer. Search of phases is carried out according to PDF-1 card file [3], then there was carried out calculation of roentgenograms according to Ritveld method with the use of FullProf program [4].

The petrographic research of samples was carried out by means of MIN-8 and Nu-2E microscopes.

The adsorption activity of crushed rock refuse is studied by means of spectrophotometric method using SPEKOL 11 device at absorption of methylene blue (MB) from the solution of initial concentration 0,01 g/l at λ = 620 nanometers.

The hydraulicity of rock refuse is determined according to the quantity of absorbed CaO. The content of active CaO in the solution in 1 and 3 days was determined by means of titration method.

III. The experimental results. *Chemical and mineralogical compositions of rock refuse.* According to the results of roentgen-phase analysis the following minerals are present in the crystal part of rock refuse: quartz SiO_2; clinochlore $(Mg,Fe)_6(Si,Al)_4O_{10}(OH)_8$ (chlorite); muscovite $K_{0.94}Na_{0.06}Al_{1.83}Fe_{0.17}Mg_{0.03}$-$(Al_{0.91}Si_{3.09}O_{10})(OH)_{1.65}O_{0.12}F_{0.23}$.

Muscovite prevails by mass, then – quartz and least of all – clinochlore. Clinochlore and muscovite refer to clay minerals which can be used in the manufacture of Portland cement clinker [5].

The wavy character of obtained diffraction patterns indicates to the presence of compounds in amorphous condition. The overall picture concerning the contents of elements in crystal and glassy state conditions is received by means of the micro x-ray analysis of samples, the results of which for noncarbonated part of rock refuse are presented in table 1. The rest of the rock refuse is composed of carbon that indicates to a small degree of rock roasting in the waste heap.

The comparison of oxide composition with the literary data for clays as the raw component of Portland cement clinker manufacture shows that in investigated rock refuse the content of oxide Al_2O_3 (1.16-20.28 % [6]) and Fe_2O_3 for «Ya. Sverdlov» mine (4.04-9.15 % [6]) is overestimated. The content of CaO, MgO and SiO_2 keeps within recommended intervals. A low content of the main (basic) oxides Ca and Mg and a big content of acid oxides Si and Al indicate the acid nature of rock refuse. The given situation does not change even after burning. According to the acidity module cemented slag refers to the ultra acid one. According to data [7] for slag of the given classification group the content of SiO_2 should be ≥ 60 %. The acidity of slag equally causes both SiO_2, and Al_2O_3.

Research of cemented samples of rock refuse. For the purpose of studying the behavior of minerals and the amorphous phase of rock refuse at high temperature they conducted cementation at t_{max} = 1580 ºC during 10 h in the oxidizing O_2 and CO_2 medium. The investigated ultra acid rock refuse form acid melts with SiO_2 content exceeding those for three-silicate O/Si+Al+Ti ≤ 2.67 [7]. With acidity increase the viscosity sharply increases and decreases the crystallization property of silicate melts. Acid melts usually solidificate that results in glass formation that is proved by thin section research at petrographic analysis.

Table 1. Relative content of elements' oxides in rock refuse of coal extraction and their classification according to the system of modules

Rock refuse of «Ya. Sverdlov» coal mine				Mass concentration, % of elements' oxides in rock refuse of «Khmelnitskiy» coal mine after burning	
Oxide	Mass concentration, % of elements' oxides			micro roentgen analysis	chemical analysis
	before burning		after burning		
	total	in big fraction			
Na_2O	0.60	–	1.099	0.670	–
K_2O	4.82	2.660	3.062	3.553	–
MgO	1.53	4.63	1.102	2.544	–
CaO	1.62	18.37	2.205	–	0.63
SiO_2	50.97	40.84	42.737	66.877	63.4
Al_2O_3	22.08	20.65	39.558	21.775	22.3
SO_3	4.85	0.857	–	–	–
Cl_2O	0.06	–	–	–	–
TiO_2	1.20	–	0.603	0.708	–
FeO	10.96	12.615	9.634 (Fe_2O_3)	3.867 (Fe_2O_3)	8.1 (Fe_2O_3)
CuO	1.12	–	–	–	–
MnO	0.19	–	–	–	–
Module	Modular classification of rock refuse				
$M_a = (Al_2O_3 / SiO_2)$	0.43	0.50	0.93	0.33	0.35
$M_s = (SiO_2 / Al_2O_3)$	2.31	2.01	1.08	3.07	2.84
$M_{al} = (Al_2O_3 / Fe_2O_3)$	2.01	1.61	4.11	5.63	2.75
$M_{cf} = (Al_2O_3+Fe_2O_3) / SiO_2$	0.65	0.81	1.15	0.38	0.48

The sample of cemented slag of «Ja. Sverdlov» mine is presented by black color slag. The sample is non-uniform according to the microstructure and consists of corundum, ferriferous spinellide (magnetite) and glass phase (fig. 1). Corundum is present in the form of grains of irregular shape of 3.2 mm size. The quantity of corundum varies in the range of 15-20 %.

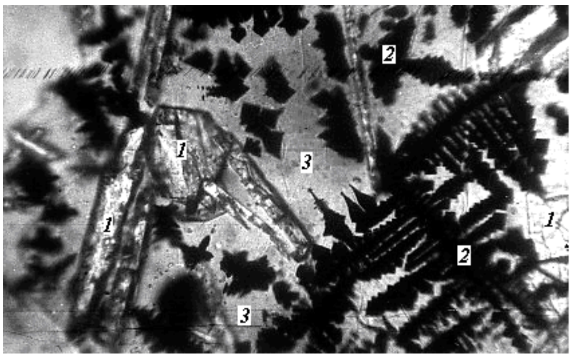
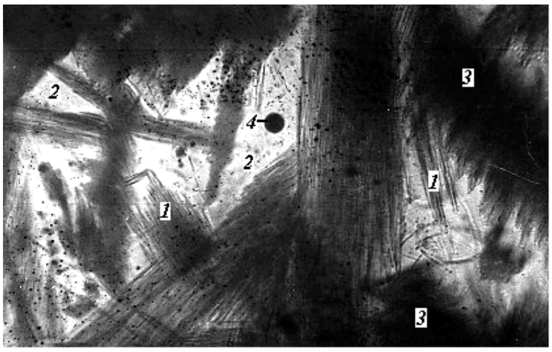


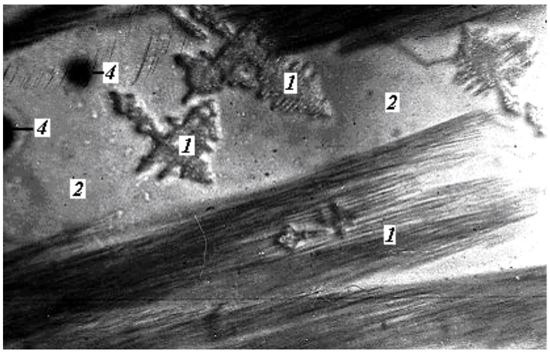
Figure 1. Sample of cemented slag of «Ya. Sverdlov» mine (X 400): 1 – corundum; 2 – magnetite; 3 – glass phase

The glass is colorless, yellowish or brownish with an average index of light refracting of $N_{aver.}$ ~ 1.525 ± 0.005.

In glass there is observed the release of ferriferous spinellide that according to the structure are close to $FeO \cdot Fe_2O_3$ magnetite that takes the form of dendrite and crystals of 10-80 microns size. There can be distinguished rare thin crystals of $3Al_2O_3 \cdot 2SiO_2$ mullite of 150 microns in length.

The sample of burnt slag of «Khmelnitskiy» mine is characterized by the presence of black color fragments with brown strips. The basic mass of the sample (fig. 2) consists of glass phase ranging from colorless to yellow-brownish one with $N_{aver.}$ ~ 1.535 ± 0.005. In glass we can observe the allocation of mullite crystals $3Al_2O_3 \cdot 2SiO_2$ to 6 mm in length at width of 4-20 microns. Mullite has refraction indicators: minimum $N_p \geq 1.658$, maximum N_g to 1.69. According to V. Lodochnikova's classification the investigated mullite refers to the fifth group of minerals according to N_p indicator, and – the sixth group of minerals according to N_g indicator. For the fifth group indicators: N_p=1.642; N_g=1.654 [8]. Slightly raised indicators of refraction and magnitude N_g-N_p = 0.032 are evidence of presence of insignificant amount of ferric oxide in the firm solution. The amount of mullite varies from 5-10% to 50-60 %.





a b

Figure 2. Sample of burnt slag of mine «Khmelnitskiy» (X 400): 1 – mullite (b – dendrite forms); 2 – glass phase; 3 – pyroxenes; 4 – metal

In some glass sections there was noticed a green-brownish allocation of pyroxenes: diopside $CaO \cdot MgO \cdot 2SiO_2$ and gedenbergite $CaO \cdot FeO \cdot 2SiO_2$. The presence of ferric spinellide ($FeO \cdot Al_2O_3$ and $FeO \cdot Fe_2O_3$) was detected as well. There was observed (<1 %) the presence of ferric release (Fe), rutile TiO_2) and hematite.

The results of petrographic research of rock refuse after clinkering are presented in table 2. The occurrence of corundum, mullite and pyroxenes is caused by the course of a number of high-temperature reactions: thermal decomposition of muscovite and clinochlore, formation of elements' oxides and their modifications; reactions between quartz and formed oxides. Possibility of formation of other compounds as primary phases can be expected as opposed to roasting of particular minerals [5].

Table 2. Results of petrographic research of clinkered samples of rock refuse

Minerals, phases	Mass fraction, %	
	«Ya. Sverdlov» mine	«Khmelnitskiy» mine
Pyroxenes	–	3-5
Ferric spinellide	10-15	5-7
Corundum (α-Al_2O_3)	30-50	–
Mullite ($3Al_2O_3 \cdot 2SiO_2$)	ghost	30-35
Glass phase	40-60	55-60

At heating to 800ºC muscovite gradually releases water with simultaneous expansion of the mineral lattice. The crystal structure collapses between 940 and 980ºC. On the basis of muscovite grains at 1000ºC there is formed γ-Al_2O_3 and a small amount of spinel, at 1200ºC there appears α-Al_2O_3 [5]. The type and quantity of polymorphic forms of Al_2O_3 depends on the temperature, duration of roasting and presence of iron impurities. At presence of ions of iron(II) and (III) the quantity of dot defects of Al_2O_3 crystal structure and its chemical activity increases.

Formation of secondary mullite can proceed at roasting from formed oxides [9] within the temperature interval of 1100-1410 ºC $3Al_2O_3 + 2SiO_2 = 3Al_2O_3{\cdot}2SiO_2$ [10].

Magnetite $FeO{\cdot}Fe_2O_3$ is formed at 1400-1600 ºC. At temperatures above 500ºC ferriferous oxides are exposed to thermal dissociation accompanied by loss of oxygen [5]. Diopside is produced at roasting and melting of the formed mix.

Application of rock refuse as perspective mineral resources. The fact of detection of mica and chlorite minerals in rock refuse is valuable. High cost of mica and difficulty of its searches and investigation of new deposits lead to the fact that mica is replaced by synthetic materials. In the case under consideration plentiful inclusions of quartz, iron and crushing of mica plates reduce the applicability of mica in electro- and radio engineering as electric insulating material.

Rock refuse of various degree of roasting is used as the component for building solutions, as raw material for manufacture of clinker brick and hydraulic binding materials [2]. The studied rock refuse cannot be used as the filling compound of concrete due to the presence of unburnt coal [11].

The affinity of chemical and mineralogical compositions of clinker Portland cement and rock refuse allows using it in cement manufacture. However it is necessary to consider a number of factors: the property of raw materials to split into oxides at rather low temperatures; lack of formation of intermediate compounds which decay with difficulty; rate of interaction of components with CaO. Some of these facts can be discussed indirectly. So, for example, the low content of alkaline metals oxides and magnesium in the investigated rock refuses will not contribute to formation at roasting of unbalanced connections complicating the formation of C_3A and C_3S. It is supposed that the rate of interaction of CaO with clay materials of rock refuse is higher than that with quartz or other slag waste [5]. High content of Fe_2O_3 promotes the occurrence of melt, at presence of which lime is better absorbed by other minerals. Joint presence of oxides Al_2O_3 and Fe_2O_3 will inevitably lead to formation of Ca alumoferrites, and with the raised content of aluminates. It, in turn, will lower the degree of thermal dissociation of Fe_2O_3, decreasing among connections $C_2F > C_6AF_2 > C_4AF > C_6A_2F > C_8A_3F$ [5] and will slow down disintegration of C_3S observed at dissolution of FeO in the lattice of C_3S.

It is possible to judge about the presence of hydraulic activity of rock refuse according to the results of estimation of absorbed lime amount CaO from 5.625% solution. Concentration of CaO was periodically controlled. Experimental results are presented in table 3.

The absorbing property of rock refuse is comparable with indicators for acid hydraulic additives and diatomite – 250-400 mg/g [12, 13]. High absorbing ability is evidence of considerable hydraulicity. Rock refuse can be used as active additives to cement clinker which should absorb not less than 50 mg/g of CaO during 30 days [6].

Table 3. Activity of rock refuse according to absorption of CaO

Rock refuse of mines	Time of interaction of slag with $Ca(OH)_2$ solution	Content of CaO in solution, %	Amount of absorption of CaO, %	Amount of absorption of CaO, mg/g
«Khmelnitskiy»	1day	3.0	2.625	261.2
	3 day	2.0	3.625	360.7
«Ya. Sverdlov»	1 day	3.625	2.0	199.0
	3 day	3.0	2.625	261.2

High hydraulicity indirectly proves to be true by determination of adsorptive activity of rock refuse. The sorption capacity of rock refuse depends on many factors: temperature of roasting, coal content, adsorptive activity of slate clay barren, etc. Direct correlation between the coal content in the rock and adsorptive activity is revealed. In the case under consideration the presence of coal particles could raise slightly the sorption capacity of rock refuse. The static exchange capacity (SEC) of rock refuse at absorption of MB was defined by means of the following formula:

$$SEC = \frac{(C_1 - C_2) \cdot V}{m}, \text{ mg/g},$$

where C_1 – initial concentration of MB, 0.01 g/l; C_2 – concentration of MB after adsorption, g/l; V – solution volume, 50 ml; m – weight of rock refuse sample, 0.5 g.

Change of optical density D of MB solution in comparison with the reference value D = 1.1 (C_{MB} = 0.01 g/l) lies in the range of 0.715-1.098. During 3 days, the optical density maximally decreases to 99.8%. According to the value of difference D the investigated rock can be referred to the group of adsorbents with high adsorptive activity. The values (SEC) obtained in the experiments are not the limiting ones; the small value of SEC is caused by the low initial concentration of MB. The efficiency value of solution sorption clearing proves the high sorption capacity of rock refuse: during 15 mines it reaches 67.5%; within 3 days it reaches the maximum value of 99%.

Hydraulic activity of rock refuse is also determined by the ratio of elements oxides, described by the system of modules (table 1). According to the value of activity module M_a the rock refuse of «Ya. Sverdlov» mine refers to the I grade of acid slag ($M_a \geq 0.4$); the rock refuse of «Khmelnitskiy» mine refers to the II grade of acid slag ($M_a \geq 0.33$).

For realization of rock refuse in manufacture of building materials, silicate module M_s should have optimum values of 1.7-3.5. The amount of Al_2O_3 release after sintering of rock refuses of «Ya. Sverdlov» mine reduces to some extent the M_s value to 1.8.

The alumina module M_{al} of rock refuse used in manufacture of Portland cement clinker, should keep within the interval of 1.0-2.5. At burning of studied rock refuse its value exceeds the border of the optimum interval, and more essentially for rock refuse of «Ya. Sverdlov» mine (to 4.11).

Classification of samples as ferriferous barren shows that all samples refer to highly active, thus the clay-ferriferous module M_{cf} > 0.45. Presumably, the most active is sintered rock refuse of «Ya. Sverdlov» mine.

High concentration of glass phase, aluminate and iron(III) oxides, high values of M_{al}, M_{cf}, high hydraulic and absorptive properties of rock refuses make possible their application at a grinding stage of clinker in manufacture of aluminous cement, as correcting or active additives to Portland cement clinker or in manufacture of calcic-slag binding material. Rock refuse of coal extraction can replace blast-furnace slag containing aluminates of Ca and Mg with concentration to 45 % Al_2O_3. As similar blast-furnace slag is melted in the limited volume its replacement is actual.

The second direction of rock refuse use as raw manufacture component of Portland cement clinker. Rock refuse is added to the raw mix before the stage of roasting for regulation of values of M_s, M_{al}, M_{cf} modules. The additive is necessary when the raw mix is characterized by adverse values of any of modules, or at manufacturing of cement clinker of particular mineralogical structure.

High content of Al_2O_3 and Fe_2O_3 oxides makes for rock refuse utilization as aluminates-ferriferous additives in manufacture of Portland clinker.

IV. Conclusions. The possibility of rock refuse utilization in manufacture of aluminous cement as correcting or active additive to Portland clinker or in manufacture of calcic-slag binding material is shown. Recycling of rock refuse will allow providing the building industry with readily available and cheap mineral raw materials and improving ecological conditions in industrial regions.

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Э. Хоботова, М. Игнатенко, Р. Маковей, В. Баумер ИСПОЛЬЗОВАНИЕ ОТВАЛЬНЫХ ПОРОД УГЛЕДОБЫЧИ В ПРОИЗВОДСТВЕ СТРОИТЕЛЬНЫХ МАТЕРИАЛОВ

Целью работы являлось изучение минералогических характеристик и гидравлической активности отвальных пород шахт «Хмельницкая» и им. Свердлова Луганской области Украины и определение возможности их использования в производстве строительных материалов. Исследован минералогический и оксидный состав отвальных пород угледобычи и их спеченных образцов. Установлена высокая гидравлическая и адсорбционная активность *отвальных пород. Показана возможность использования отвальных пород в качестве корректирующей добавки в производстве алюминатного цемента или активной добавки к портландцементному клинкеру или в производстве известково-шлаковых строительных материалов. Утилизация отвальных пород обеспечит строительную отрасль доступным и дешёвым сырьём и улучшит экологические условия промышленных регионов.*

Ключевые слова: *отвальная порода, угледобыча, строительные материалы, вяжущие материалы*

Е. Хоботова, М. Ігнатенко, Р. Маковей, В. Баумер ВИКОРИСТАННЯ ВІДВАЛЬНИХ ПОРІД ВУГЛЕВИДОБУТКУ В ВИРОБНИЦТВІ БУДІВЕЛЬНИХ МАТЕРІАЛІВ

Метою роботи було вивчення мінералогічних характеристик і гідравлічної активності відвальних порід шахт «Хмельницька» та ім. Свердлова Луганської області України і визначення можливості їх використання у виробництві будівельних матеріалів. Досліджений мінералогічний і оксидний склад відвальних порід вуглевидобутку та їх спечених зразків. Встановлена висока гідравлічна і адсорбційна активність відвальних порід. Показана можливість використання відвальних порід як коректуючої добавки у виробництві алюмінатного цементу або активної добавки до портландцементного клінкеру або у виробництві вапняно-шлакових будівельних матеріалів. Утилізація відвальних порід забезпечить будівельну галузь доступною і дешевою сировиною та покращить екологічні умови промислових регіонів.

Ключові слова: *відвальна порода, вуглевидобуток, будівельні матеріали, в'яжучі матеріали*

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ВИТЯГ ЦИНКУ ЛУГОМ З ВІДХОДІВ ГАЛЬВАНИЧНОГО ВИРОБНИЦТВА

Досліджено процес витягу цинку з відходу гальванічного виробництва. Визначений склад вихідної сировини, запропоновані стадії її попередньої обробки: термічна, промивка водою, видалення заліза та міді. Розглянута можливість витягу цинку з отриманої концентрованої сировини. Вивчено вплив температури, концентрації лугу і інших факторів на ефективність витягу.

Ключові слова: *відходи гальванічного виробництва, цинк, кадмій, нікель, кобальт, термічна обробка, промивання водою, амфотерність, витяг розчином лугу.*

Утилізація відходів гальванотехніки, яка охоплює широко розповсюджені процеси нанесення захисних покрить на поверхню різних металевих виробів, є досить складною задачею [1-4]. В гальванотехніці під час таких процесів як цинкування, нікелювання та кадмування, утворюються відходи з низьким значенням pH, які містять в своєму складі цінні компоненти, а саме цинк, нікель, кадмій та інші. Для зменшення негативного впливу на довкілля цих відходів їх