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MINERAL COMPOSITION OF DUMP BLAST FURNACE SLAG

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***Abstract*.** Industrial wastes, accumulating in a dumping ground, have useful technical properties in many cases, so they can be considered as secondary resources. The investigation of slag properties and modifications in different conditions needs a complex approach that includes X-ray phase, electron microscopic and petrographic analyses. The research aim is to substantiate the resource value of Zaporozhstal PJSC dump blast furnace slag on the basis of chosen experimental methods. X-ray phase analysis allows us to discover the minerals of blast furnace slag that are crystalline: rankinite $3CaO{\cdot}2SiO_2$, quartz SiO_2, helenite $2CaO{\cdot}Al_2O_3{\cdot}SiO_2$, bredigite α-$2CaO{\cdot}SiO_2$, okermanite $2CaO{\cdot}MgO{\cdot}2SiO_2$ and pseudowollastonite α-$CaO{\cdot}SiO_2$. The minerals okermanite, bredigite and pseudowollastonite are technically useful to produce binders as they are hydraulically active. The mass fraction of a vitreous component, which composes half of blast furnace slag mass of Zaporozhstal PJSC, was computed. Amorphous phases testify on the higher sorption and chemical slag activation that are important in terms of the use of slag to produce binders. The mass contribution of amorphous substance state is slightly higher in large fraction slag. Microphotographs of the surfaces of blast furnace slag particles show high loosening degree and needle-shaped and lamellar crystallines that stipulate sorption properties of the slag. The dump blast furnace slag of Zaporozhstal PJSC can be recommended to produce binders – Portland cement and Portland slag cement – at totality of chemical parameters: high concentration of hydraulically active minerals and amorphous phase, highly developed surface of slag particles and surface sorption activation.

Keywords: furnace slag, chemical composition, minerals, amorphous phase, sorption properties, hydraulic activity, particle surface morphology, binders.

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Introduction

A considerable amount of slag is formed as wastes or by-products in the metallurgical industry. As industrialization grows, available ground to dump large volumes of metallurgical slag decreases and the cost of utilization goes up. Metallurgical and blast-furnace slag contains rather high amount of precious metals and minerals. However, at present, most industrial slag is utilized rather than used, taking into account its useful properties. This approach can lead to a considerable economic profit, resource and energy saving and it can reduce emissions in metallurgy. Slag is processed using such mechanical and physicochemical methods as crushing, grinding, hydrocycling, magnetic separation, flotation, leaching and roasting [1]. The most economical and effective option to reduce metallurgical waste is to recycle it in order to considerably decrease CO_2 emission that is characteristic of slag [2]. Processing of slag from one form to another to reuse it at the same enterprise or in different industrial units is very important to save metals and mineral resources as well as to protect the environment.

Last years the ecological aspect is imperative to control industrial wastes. The technologies which put into practice the industrial ecology concept enter the market if ecological and economic profits are considerable. This paper analyses modern innovative technologies. Metallurgical slag of various metallurgical processes is used in different ways depending on slag characteristics. The use of metallurgical slag in the construction industry increases in value because natural resources deplete. The slag of cast iron and steel is often used in construction engineering. Slag-based building material properties vary if slag is received from different technological processes. A ne-cessary condition to use slag is a preliminary investigation of its physicochemical and mechanical properties. The authors of paper [3] have researched the slag properties, hydration, reactivity, hydraulic activity, pH, glass content and microstructure. Paper [4] shows the possibility to use the slag of stainless steel production to brickwork after preliminary carbonization and thermo-alkaline activation. It is proposed to use ladle furnace slag (by-product of secondary steel refinement) [5] as a binder in building mortars to replace hydraulic lime. Basic oxygen furnace slag aggregates are recommended [6] as a binder in slag-bituminous mixtures. In this case, a disadvantage is a low content of C_3S and C_2S minerals in slag; it makes slag a poor hydraulic binder.

Three main directions to use slag in binder production are: partial substitution of Portland cement (PC), i. e. creation of Portland slag cement (PSC); development of new slag-alkali binders (SAB) and the use of slag as raw material to produce PC. In the last case, slag minerals disintegrate under high temperature roasting and the oxi-des are used to make new minerals that are characteristic of PC.

Papers [7 – 10] consider some options to use metallurgical slag in PSC. Putting it into practice reduces a need in PC that considerably decreases pollution and paves the way for cleaner and more economical building materials. Research [10] shows that the development of stress in PSC in the early phases of hardening is tied to the low value of the MgO/Al_2O_3 ratio in granulated blast furnace slag. Papers [11 – 15] analyze the impact of temperature, slag content, conditions of hydration and activation on PSC properties. A slag dosage of up to 70 % in a binder contributes to the active compression and bending strengthening of concrete especially in the later phases of maturation. In addition, slag activates clinker mineral hydration [15].

The second direction of the use of blast-furnace slag in the production of building materials is the creation of SAB. The authors of paper [16] have determined the high values of compression and tension strength for the following composition: 50 % of blast-furnace gravel, 50 % of granulated blast-furnace slag and 18 % of liquid glass as hardening activator. Papers [17, 18] also show that low active blast-furnace slag and the slag of other origin having low hydraulic activity can be used to produce SAB. The advantages of this slag application are practically complete binding of slag heavy metals by various alkaline agents and the stoppage of their migration into the environment.

The putting of steel production slag into raw meal to produce Portland cement clinker shows that the use of slag has no impact on the mineralogical characteristics of PC. Hydration products have been investigated using the X-ray diffraction analysis for 90 days running [19].

The issue of completeness of preliminary obtained scientific data that characte-rise technical properties and slag chemical composition, that's, the issue of the sequence of slag research methods, is disputable. It is proposed [20] to research various types of steel slag, which is formed in the course of steel smelting in oxygen and electric arc furnaces and refinement, using the methods of X-ray diffraction analysis and scanning electronic microscopy. The approaches to analyze the chemical and mineral components of converter steel slag are similar: electron microscopy, energy spectrum analysis and X-ray diffraction analysis [21]. The chemical and mineral composition of steel slag stored in utilization areas is determined by the methods of elemental chemical analysis, X-ray diffraction, thermal analysis, scanning electron microscopy and FTIP spectroscopy [22].

We propose the method [23] to determine an industrial waste resource value in order to utilize waste as technical materials; it optimizes research sequence and increases its effectiveness and completeness in terms of the determination of some waste properties. The choice of research methods is based on the need to investigate the mineral, elemental, oxidation and radionuclide composition of industrial waste, the structure of its surface, sorption and hydraulic activation. The used research methods are as follows: X-ray and phase analysis, gamma-ray spectrometric analysis, electron probe microanalysis and petrographic analysis.

The objective of this research is to substantiate the resource value of dump waste blast furnace slag on the basis of chosen experimental methods at Zaporozhstal PJSC.

Research methodology

Slag samples are taken using the rules in Recommendations [24]. Sieving to get granulometric fractions is performed using a set of sieves. The fractions obtained are as follows, mm: >20, 10 – 20, 5 – 10, 2.5 – 5, 1.25 – 2.5, 0.63 – 1.25, <0.63. The granulometric composition of dump blast furnace slag is shown on the Fig. 1.

The mineral composition of a slag crystalline component is determined using X-ray phase analysis [25] that is carried out with Siemens D500 powder diffractometer through copper radiation with a graphitic monochromator. Full profile diffraction patterns are measured within a range of angles 5° < 2θ < (110 – 120°). The primary search of phases is carried out using the PDF-1 card file [26] and then the computation of X-ray patterns is made using Rietveld method and FullProf program [27].

Petrographic analysis of the crystalline and amorphous components of dump blast furnace slag is made with MIN-8 and Nu-2E microscopes in transmitted light in immersion preparations and transparent slices.

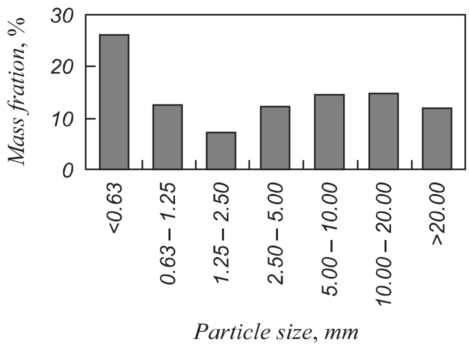

Fig. 1. Granulometric composition of dump blast furnace slag of Zaporozhstal PJSC

Рис. 1. Гранулометрический состав отвального доменного шлака ПАО «Запорожсталь»

The particle surface morphology is determined by means of the method of electron probe microanalysis by JSM-6390 LV scanning electron microscope.

Mineral composition of slag crystalline component

Results of X-ray phase analysis

According to the diffraction patterns, six phases are determined: rankinite $Ca_3Si_2O_7$ ($3CaO{\cdot}2SiO_2$), quartz SiO_2, helenite $Ca_2Al(Al, Si)_2O_7$ ($2CaO{\cdot}Al_2O_3{\cdot}SiO_2$), bredigite $Ca_{14}Mg_2(SiO_4)_8$ (α-$2CaO{\cdot}SiO_2$), okermanite $Ca_2MgSi_2O_7$ ($2CaO{\cdot}MgO{\cdot}2SiO_2$), and pseudowollastonite $CaSiO_3$ (α-$CaO{\cdot}SiO_2$). Mineral mass fractions are in described in Table 1 for three groups of particles: small (<0.63 mm), medium (0.63 – 5.0 mm), large (>5.0 mm).

Petrographic analysis has confirmed most of the phases and has discovered some phases. Comparative analysis of the mineralogical compositions of slag separate fractions carried out by two experimental methods is considered in Table 1.

According to the results of X-ray phase analysis, the mass fraction of minerals rankinite and okermanite decreases as sifted grain size increases, content of helenite slightly increases, bredigite is mainly in a large fraction. The content of pseudowollastonite depends on the sample particle size very much. The maximum content of this mineral falls on medium-sized slag particles.

Petrographic research results

Petrographic analysis has been applied to investigate two groups of slag particles according to their dispersity (small and large) (Table 1). Slag glass can contain crystallites, which are the germs of the crystallines of slag minerals.

Group of large slag particles contains the light grey fragments of 2 – 4 mm in size (in the centre of it they are darker). The sample predominantly consists of crystalline phases. They are pseudowollastonite, melilite, pyroxene, β-$2CaO{\cdot}SiO_2$. Pseudowollastonite (α-$CaO{\cdot}SiO_2$) is elongated prismatic crystallines, sometimes subparallel, sometimes intersecting. Crystalline length is up to 300 μm and

Table 1

Mineral composition of granulometric fractions of dump blast furnace slag of Zaporozhstal PJSC

Таблица 1. **Минеральный состав гранулометрических фракций отвального доменного шлака ПАО «Запорожсталь»**

Phase	Results of X-ray phase analysis Particle size group of slag						Phase	Results of petrographic analysis of minerals in groups of slag by dispersibility, mass fraction (%)	
	small		medium		large			small	large
	Mass fraction, %	Particle size, nm	Mass fraction, %	Particle size, nm	Mass fraction, %	Particle size, nm			
SiO_2	6	>500	5	257	4	57	SiO_2	5 – 10	–
α-$2CaO{\cdot}SiO_2$ bredigite	5	57	8	78	28	71	β-$2CaO{\cdot}SiO_2$	10 – 15	19
$2CaO{\cdot}Al_2O_3{\cdot}SiO_2$ helenite	32	>500	33	>500	40	68	melilites: helenite + okermanite	25 – 30	34
$2CaO{\cdot}MgO{\cdot}2SiO_2$ okermanite	7	126	4	107	2	120			
α-$CaO{\cdot}SiO_2$ pseudowollastonite	14	29	18	22	10	24	α-$CaO{\cdot}SiO_2$ pseudowollastonite	15 – 20	17
$3CaO{\cdot}2SiO_2$ rankinite	36	90	25	94	12	84	pyroxenes: $CaO{\cdot}MgO{\cdot}2SiO_2$ diopside + $CaO{\cdot}FeO{\cdot}2SiO_2$ hedenbergite	5 – 10	7
							$CaCO_3$ calcite	10 – 15	2
							CaS oldhamite	1 – 2	1
							Glass phase	10 – 15	8

width is 4 – 9 μm (Fig. 2). β-$2CaO{\cdot}SiO_2$ forms the isometric crystallines of 20 – 50 μm.

Melilites (solid solution "helenite $2CaO{\cdot}Al_2O_3{\cdot}SiO_2$ – okermanite $2CaO{\cdot}MgO{\cdot}2SiO_2$") are prismatic crystallines that are maximum as long as 200 μm and as wide as 8 – 30 μm. Taking into account the average refractive index value $N_{av.} \sim 1.654$, melilites contain approximately equal quantities of helenite and okermanite.

Pyroxenes (solid solution "diopside $CaO{\cdot}MgO{\cdot}2SiO_2$ – hedenbergite $CaO{\cdot}FeO{\cdot}2SiO_2$") are prismatic greenish-brownish crystallines of up to 20 – 35 μm in length. Taking into account the refraction coefficient, the hard mixture contains approximately equal quantities of diopside and hedenbergite.

Crystalline phases are cemented by the finest films of a glass phase. A comparison between the glass phase refractive index $N \sim 1.60 – 1.63$ and the Portland cement glass refractive index $N = 1.71 – 1.90$ [28] indicates their considerable difference.

The group of small particles of slag compositionally and structurally is similar to the group of large particles but differs from it in terms of quartz fragments and a larger content of calcite. The portlandite $Ca(OH)_2$ might be available in the form of small (≤ 4 μm) grains in fine-grained aggregates with calcites.

The results of X-ray phase analysis and petrographic analysis demonstrate a similarity in discovering certain minerals and their quantitative contribution (Table 1). The two methods discover quartz, $2CaO{\cdot}SiO_2$, pseudowollastonite and melilites. The two methods to determine the qualitative mineral composition of slag have some differences. X-ray phase method discovers rankinite having a high mass fraction in a crystalline phase. Petrographic analysis additionally determines pyroxenes: diopside, hedenbergite, calcite, oldhamite and a glass phase. The mass fraction of a glass phase is 10 – 15 %, which is within the Portland cement glass concentration interval of 3 – 25 % [28]. $2CaO{\cdot}SiO_2$ is discovered in various modifications. A chemically active α-modification is found during X-ray phase research.

X-ray phase analysis determines the mass fraction of minerals in a crystalline fraction component only; petrographic analysis determines the mass fraction in the whole fraction. Quartz SiO_2 is not discovered in large slag particles by the petrographic analysis. It seems to be absent in a glass phase. According to the results of X-ray phase analysis, the mass contribution of SiO_2 decreases if the size of slag particles increases. For large slag particles it is 4 % only (Table 1). X-ray phase analysis shows higher values of total content of helenite and okermanite than petrographic analysis does. It definitely testifies the absence of melilites in the amorphous part of fractions. Besides, there are discrepancies in terms of the ratio of helenite to okermanite in both methods. Petrographic analysis shows their equal amount but X-ray phase analysis indicates a considerable predominance of helenite.

The mineral composition of crystalline part of dump blast furnace slag testifies that it is kindred to the mineral composition of granulated slag. The content of hydraulically active minerals – bredigite, okermanite, pseudowollastonite – increases if slag particle becomes larger and reaches its maximum value of 40 % for large slag particles. Availability of these mineral phases testifies about suitability of slag to produce binders.

Mass fraction of crystalline and amorphous substances in dump blast furnace slag

The presence of amorphous substances is confirmed by evident wavy background in the X-ray diffraction patterns of some slag samples. Therefore, computation of the mass fraction of crystalline and amorphous substances is performed for dump blast furnace slag fractions of Zaporozhstal PJSC. The method mentioned in [29] is

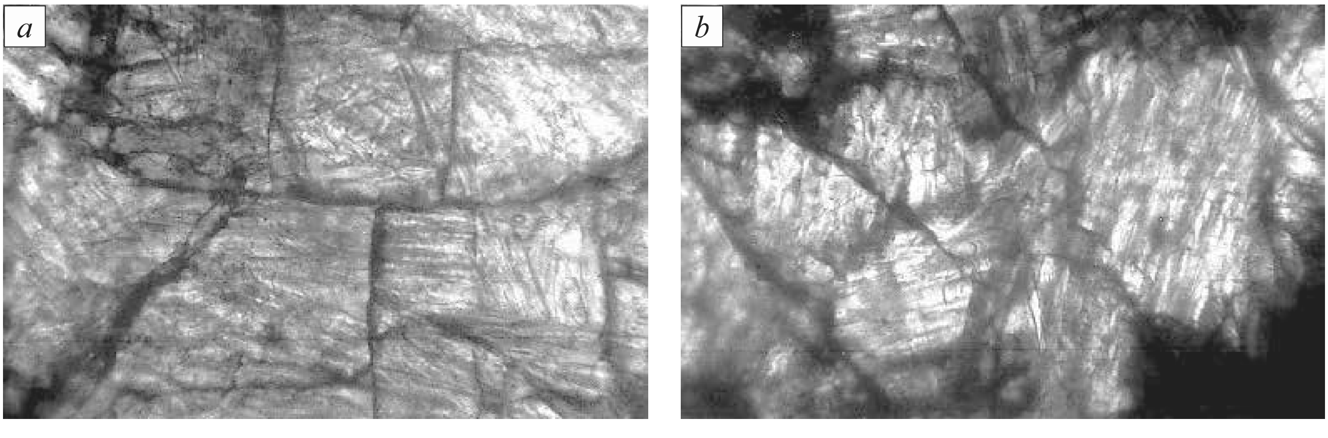

Fig. 2. Microphotographs of a sample of over 20 mm fraction of dump blast furnace slag of Zaporozhstal PJSC (×500):
a – parallel nicols; *b* – crossed nicols

Рис. 2. Микрофотографии образца фракции более 20 мм отвального доменного шлака ПАО «Запорожсталь» (увеличение 500):
а – николи параллельные; *б* – николи скрещенные

Table 2

Calculation results of average mass fraction of a substance in crystalline and amorphous state

Таблица 2. **Результаты расчета средней массовой доли вещества в кристаллическом и аморфном состоянии**

Substance state	Mass fraction (%) of various states of a substance in groups of slag by dispersibility	
	small	large
Crystalline	48 (46 – 50)	43 (41 – 45)
Amorphous	53 (51 – 55)	58 (56 – 60)

used to calculate it. The computation results are considered in Table 2.

An amorphous mass fraction is high if we take into account that slag is dump waste, i. e. it is slowly cooled. Slag is half amorphous substance. It confirms the possibility of adsorption of strange ions and compounds by means of a sorption active slag surface. Amorphous phases testify about higher sorption and chemical slag activation; it is important in terms of using slag to produce binders. The mass fraction of an amorphous substance is slightly higher in a large slag fraction.

Particle surface morphology

The sorption activation of a particle surface is usually characterised by their surface morphological features and it increases if a loosening degree rises. The microphotographs of blast furnace slag grain surfaces (Fig. 3) testify that the particle surface of the fraction of a particle size less than 0.63 mm has a high degree of loosening. The fraction 2.5 – 5.0 mm is characterised by needle-shaped structures with high sorption capacity. The fraction particles with a particle size more than 20 mm have lamellar structure that contributes to a sorption decrease.

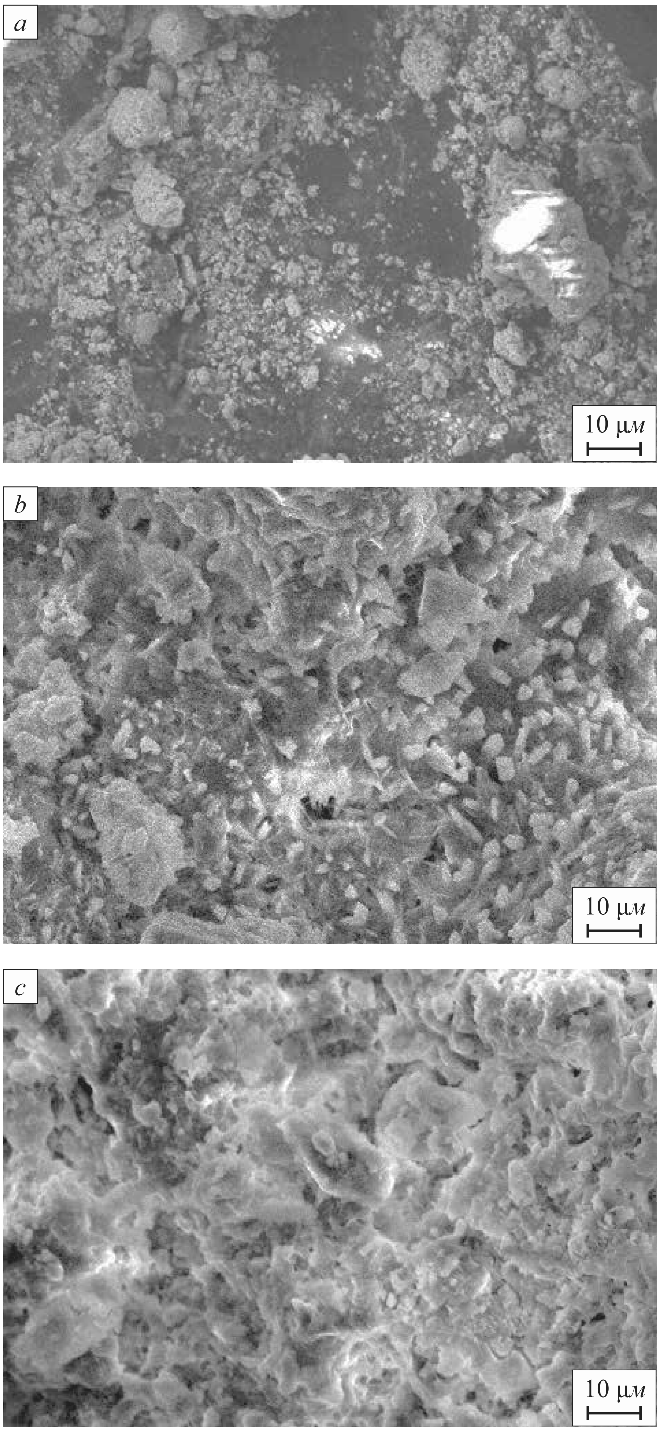

Fig. 3. Micrographs of the particles' surface of dump blast furnace slag of Zaporozhstal PJSC of fractions: less than 0.63 mm (*a*), of 2.5 – 5.0 mm (*b*); over 20 mm (*c*)

Рис. 3. Микрофотографии поверхности частиц отвального доменного шлака ПАО «Запорожсталь» фракций менее 0,63 мм (*а*), 2,5 – 5,0 мм (*б*); более 20 мм (*в*)

Conclusions

The choice of physicochemical research methods, which enable us to correctly determine the mineral composition of dump blast furnace slag and to forecast its resource value, is substantiated. It is shown that in various situations the investigation of slag properties and modifications needs a complex approach that includes X-ray phase, electron microscopic analyses and petrographic research. X-ray phase and petrographic analyses enable us to discover the crystalline minerals of blast furnace slag and to confirm the amorphous state of substances. It is proved that slag contains minerals that are technically valuable to produce binders. The glass component mass fraction, which composes half of Zaporozhstal PJSC blast furnace slag mass, is computed. The dump blast furnace slag of Zaporozhstal PJSC can be recommended to produce binders – Portland cement and Portland slag cement – at totality of chemical parameters: high concentration of hydraulically active minerals and amorphous phase, highly developed surface of slag particles and surface sorption activation.

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