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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

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ANALYSIS AND PROSPECTIVE UTILIZATION OF TECHNOGENIC SLAG WASTE FROM A LEAD PLANT

Abstract. The article presents the analysis and results of the study of technogenic slag waste of lead-zinc production. Slags of lead-zinc production contain a large number of toxic compounds: lead, zinc, osmium, cadmium, which are dangerous sources of environmental pollution. Due to the open storage of slags, it was found that the maximum permissible concentrations of lead were exceeded. Utilization of man-made slag waste is of great importance for reducing the negative impact on the safety of life and improving the environmental situation in the region. At the same time, slags are valuable raw materials containing compounds of non-ferrous and rare-earth metals.

The article shows the results of laboratory studies of slags to determine the qualitative and quantitative composition of valuable components in the waste of lead production and the possibility of their further processing and disposal. Studies of the material of the heavy slag fraction were carried out on an electron probe microanalyzer of the JEOL IXA-8230 Electron Probe microanalyzer brand. X-ray diffractometric analysis of the average slag sample was performed on a DRON-4 diffractometer with Cu radiation, graphite monochromator. Samples were selected heavy fraction and manufactured artificial polished sections (briquettes). The sections were studied under the microscope of the brand LEICA DM 2500P and immersion in liquids.

According to the results of research, it was found that lead slags contain a sufficiently high amount of non-ferrous metal compounds: lead oxide up to 0.7 % and zinc oxide up to 8.5 % of the weight amount of slag, which makes the process of recycling toxic waste from lead production technically and economically feasible.

Keywords: waste, lead-zinc waste, secondary raw materials, non-ferrous metals, valuable components, waste processing, waste disposal, low-waste and non-waste technologies.

Introduction. An important area of environmental protection in metallurgy is the introduction of waste-free technologies and technologies for the integrated use of raw materials. This ensures the enrichment of ores, the rational completeness of the extraction of the main and related elements, and the disposal of production waste without harming the environment.

Due to various economic reasons, the Shymkent lead Plant was finally shut down in 2011. But nevertheless, the environmental situation of the city is still negatively affected by the accumulated waste in the form of slags in the amount of approximately 2 million tons, which is stored in open storage under the influence of sunlight, air oxygen and atmospheric precipitation [1-4]. Lead plant slags are an important raw material containing various non-ferrous metals, and currently non-ferrous metals obtained from secondary raw materials play an important role in the overall balance of production and consumption of non-ferrous metals in the Republic of Kazakhstan: their share in relation to the total production of non-ferrous metals is about 25%. For example, according to the authors [5-8], lead has a high economic value and the scope of use of lead has changed in recent years, and now approximately 80% of global consumption is accounted for by the sector of production of electric batteries. The pliability, density and anti-corrosion properties of lead are still actively used in the construction of tanks for storing caustic liquids and as protection against X-rays and radiation. Lead is used in the manufacture of paints and pigments and other chemical compounds. Lead-acid batteries are used in cars. The reason for the widespread use of lead in batteries in automotive and industrial engineering is that lead is able to provide a large amount of electricity for a short period of time, which is necessary to power the vehicle's starter motor [9-11].

Batteries are also used to provide current, to drive heavier vehicles such as diesel or electric locomotives and submarines, and as a source of spare energy in installations with critical functions such as telecommunications facilities and hospitals. Lead has the highest utilization rate in secondary processing of all metals. Lead is used in the chemical industry, as a component in the production of reagents and paints, as well as in the IT sector, where the metal is used as solders and additives. Due to its unique physical and chemical properties, lead has found a place in the production of various engineering products, such as protective coatings for buildings and structures. High corrosion resistance of the metal, durability and ease of use are the main advantages when using it), as well as for use in medical devices for protection against gamma radiation, in the production of X-ray and spectrographic equipment. Lead is a part of bronzes, brasses, babbits, and typographic alloys [12-14].

Another non-ferrous metal proposed for extraction in the process of recycling lead production slags is zinc. Zinc is used for galvanizing metal products, in order to give them anti-corrosion properties, and in this regard, the demand for zinc remains high, due to the rapid growth in the production of anti-corrosion coatings. Zinc is also used in the production of alloys (brass, nickel silver), printing materials. Zinc compounds are also used in the production of pigments for paints, the production of rubber, glass and glaze. Another important field of application is in the composition of neutralizing cosmetic pastes and pharmaceutical preparations [15].

Thus, the most economical and effective method to reduce the negative impact of dumps on the ecological situation of the city is its further processing. Even after the extraction of non-ferrous metals, slags can be used in the production of cement and building materials, since slags of lead production contain up to 75-85% iron, calcium, and silicon oxides [16].

Problem statement. The research object is lead-containing slag dumps from the lead plant, which are production costs. To determine the methods of disposal and processing of lead slags for extraction of zinc oxide, lead oxide, there is a number of scientific works based on the need to determine the chemical composition and quantitative content of non-ferrous metals and other compounds. The main goal of the research is to create a highly efficient technology for processing the lead production slags, which allows to involve the lead production slag wastes in processing as secondary resources. This, in turn, will allow to rationally use natural resources and reduce the area occupied by the wastes.

Preliminary data on the lead production slag obtained in the production cycle showed that the lead production wastes are smelter slags. The particles of lead-containing slags are in the form of irregular granules, the material density in the loose body is 2 t/m^3, the angle of repose is approximately 35°, the particle size mainly ranges from 2-6 mm, and there is a small number of particles of about 10 mm. The lead production dry slag components are shown in table 1.

Table 1 - Components of slag of lead-zinc production in a dry state

Element	Pb	Zn	Cu	Fe	SiO_2	CaO	K_2O	S	O	Other	Total
Numerical value	2.38	9.81	0.97	25.31	24.62	16.21	1.42	1.35	10.16	7.32	100

Note: The numerical value of a sample is the average of a randomly selected sample

To determine the chemical composition of the lead production slags, we performed spectral, X-ray phase, thermal and chemical analyses. The studies were carried out at Institute of Metallurgy and Ore Beneficiation of the National Academy of Sciences of the Republic of Kazakhstan, Almaty, and at K.I. Satpayev Institute of Geological Sciences.

The study of the material composition was carried out on bulk slag material, externally black, with the particle size of 2 to 6 mm. A heavy fraction was isolated from the sample, according to which artificial polished sections (briquettes) were made. The polished sections were examined under LEICA DM 2500P microscope. Along with this, the sample was studied under a microscope in immersion liquids, and as a result, samples were selected for further research.

X-ray diffractometric analysis of the slag samples was carried out on DRON-4 diffractometer with Cu-radiation, graphite monochromator. Conditions for recording diffraction patterns: U=35kV; I=20mA; scale: 2000 imp; time constant 2s; shooting theta – 2 theta; detector 2 deg/min. Semiquantitative analysis was carried out on the basis of diffraction patterns of the powder sample using the method of equal weights and artificial mixtures. The quantitative ratios of the crystalline phases were determined. The diffraction patterns were interpreted using ASTM Powder diffraction file and diffraction patterns of minerals free of impurities. The contents were calculated for the main phases. Possible impurities, the identification of which cannot be unambiguous due to low contents and the presence of only 1-2 diffraction reflections or poor crystallization, are indicated in table 2.

Table 2 – The results of the semiquantitative atomic emission spectral analysis of the slag's technological sample

Elements	Content of elements, %	Elements	Content of elements, %
Gold	<0.0002	Silver	0.001
Silicon	>>1.0	Magnesium	>1.0
Aluminum	>1.0	Calcium	>1.0
Copper	0.3	Rhenium	<0.0003
Nickel	0.0025	Chromium	0.015
Antimony	<0.002	Cobalt	0.005
Arsenic	<0.01	Molybdenum	0.01
Iron	>>1.0	Strontium	0.1
Manganese	0.2	Tellurium	<0.003
Titanium	0.3	Lanthanum	0.002
Zinc	>1.0	Bismuth	0.0005
Potassium	<1.0	Beryllium	0.0003
Sodium	>1.0	Zircon	0.01
Tin	0.001	Ytterbium	0.0002
Barium	0.3	Yttrium	0.003
Scandium	0.0005	Antimony	0.07
Vanadium	0.007	Cerium	0.005
Wolfram	0.005	Gallium	0.002
Germanium	0.001	Thallium	<0.0005
Cadmium	<0.0005	Lead	0.1
Iridium	<0.001	Niobium	<0.001
Arsenic	<0.01	Mercury	<0,003
Platinum	<0.001	Palladium	<0.0002
Rhodium	<0.0005	Ruthenium	<0.001

As follows from the data of X-ray diffractometric analysis presented in table 2, the slag samples are represented by amorphous phases of composition close to crystalline phases of natural origin, namely fayalite, wollastonite, zinc oxide and iron oxide. In an immersion preparation in transmitted light under a microscope, all these phases are externally black and amorphous, however no crystalline formations are observed [17]. The identification of mineral phases according to the data of X-ray diffractometric analysis is shown in the diffractogram in figure 1.

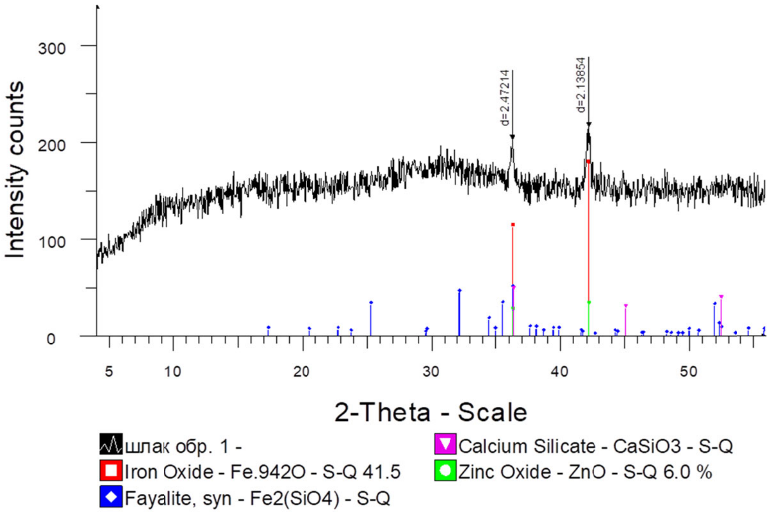

Figure 1 - Diffractogram of the slag sample

The results of interplanar distances and phase composition of the slag sample are presented in table 3.

Table 3 – The interplanar distances and phase composition of the slag sample

Slag sample		
d, Å	I %	Phase
2.47214	93.8	Iron Oxide, Fayalite, Zinc Oxide, Calcium Silicate
2.13854	100.0	Iron Oxide, Zinc Oxide

All presented diffraction peaks shown in table 3 belong only to the above phases. The characteristic diffraction reflections are noted, which allow to identify the phases present.

The results of the semiquantitative X-ray phase analysis of the crystalline phases are shown in table 4.

Table 4 – The results of the semiquantitative X-ray phase analysis of the crystalline phases

Mineral phase	Chemical formula	Content, %
Iron Oxide	$Fe_{0.942}O$	41.5
Fayalite, syn	$Fe_2\ (SiO_4)$	35.4
Calcium Silicate	$CaSiO_3$	17.2
Zinc Oxide	ZnO	6.0

The analysis of table 3 shows that the slag sample is based on an amorphous substance with the listed crystalline phases with superimposed reflections.

When examining the sample in the polished briquette in the reflected light, shown in figure 1, it was revealed that the slag sample consists of an amorphous matrix with numerous inclusions of heterogeneous copper mineral phases, which externally resemble natural copper sulfide minerals such as chalcopyrite and even native copper. They often have a rounded isometric outline and a light yellow color, typical of chalcopyrite.

In order to identify industrially valuable slag minerals, studies of the material of the heavy slag fraction were carried out on an electron probe microanalyzer. To do this, the surface of the polished briquette was carefully scanned, which allowed us to detect copper mineral phases in the sample, as well as the accompanying artificial lead-zinc mineral formations and study their composition. The research was carried out on a modern electronic microanalyzer brand JEOL IXA–8230 Electron Probe microanalyzer.

In order to study the composition of the sample matrix and very small inclusions, an electron probe scan of the plane of the polished briquette was performed (figure 2, 3, 4).

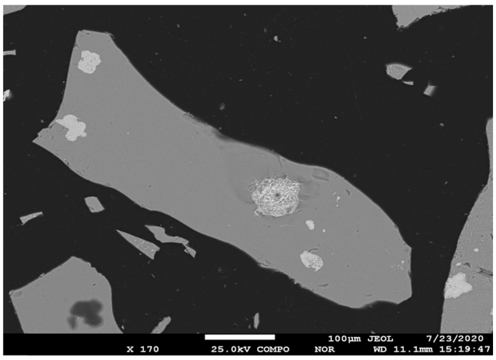

Figure 2 - Mineral matrix with inclusions of copper mineral phases

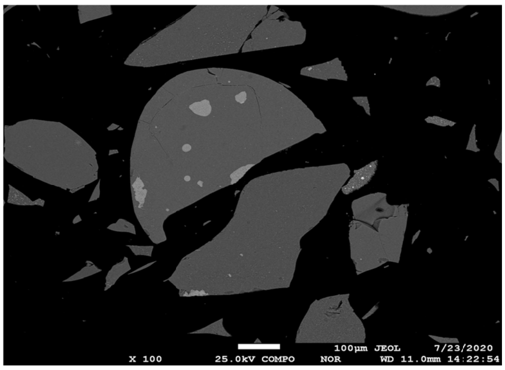

Figure 3 - Electron-probe inclusions of complex copper mineral phases in the matrix

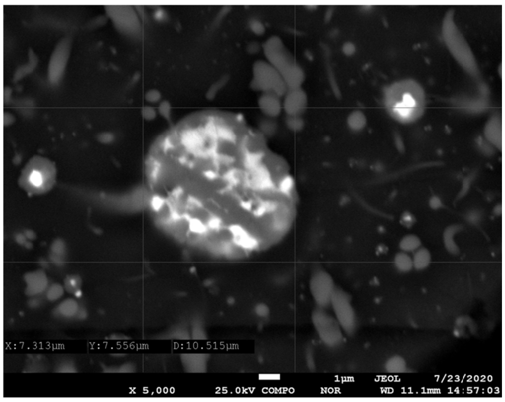

Figure 4 - Matrix with inclusions of oxides of complex lead-zinc-ferruginous-copper mineral phases

Conclusion. The results of spectral, X-ray phase, electron-probe and chemical analyses showed that the slag of lead production has a sufficiently high number of non-ferrous metal compounds: the content of lead oxide up to 2 %, zinc oxide up to 17 % and copper oxide up to 1.25 % of the total weight of the sample. The qualitative composition and content of non-ferrous metals of lead slags makes it possible to make the process of disposal of toxic waste of lead production technically and economically feasible.

By scanning the heavy fraction of the slag sample on an electron probe microanalyzer, the following useful components were identified: copper in the form of sulfides, complex compounds of lead, zinc, iron and copper oxides, which are found as inclusions in an amorphous host matrix of complex composition.

The results of preliminary tests allow us to choose a technology for more complete and selective extraction of lead and zinc oxides from the slag waste of lead production. When using a selective method for extracting non-ferrous metals, it is expected to improve the ecological state of the environment and reduce the negative impact on human health due to the disposal of toxic slags from lead production. At the same time, a significant contribution is made to the development of the system of rational use of natural and secondary resources.

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ҚОРҒАСЫН ЗАУЫТЫНЫҢ ТЕХНОГЕНДІ ҚОЖ ҚАЛДЫҚТАРЫН ТАЛДАУ ЖӘНЕ КЕЛЕШЕКТЕ КӘДЕГЕ ЖАРАТУ

Аннотация. Мақалада қорғасын-мырыш өндірісінің техногенді қож қалдықтарын зерттеу нәтижелері мен талдауы берілген. Қорғасын-мырыш өндірісінің шлактарында көптеген улы қосылыстар бар: қорғасын, мырыш, осмий, кадмий, олар экологиялық ластанудың қауіпті көзі болып табылады. Қождарды ашық сақтауға байланысты қорғасынның шекті рұқсат етілген концентрациясының асып кетуі анықталды. Техногенді қож қалдықтарын пайдаға асыру аймақтың экологиялық жағдайын жақсарту және өмір тіршілігінің қауіпсіздігіне теріс әсерін төмендету үшін үлкен маңызға ие. Сонымен қатар, шлактар түсті және сирек кездесетін металдардың қосылыстары бар құнды шикізат болып табылады.

Мақалада қорғасын өндірісінің қалдықтарындағы құнды компоненттердің сапалық және сандық құрамын анықтау бойынша қождарды зертханалық зерттеу нәтижелері және оларды әрі қарай өңдеу мен кәдеге жарату мүмкіндігі көрсетілген. JEOL IXA-8230 Electron Probe microanalyzer маркалы электронды зондты микроанализаторда қождың ауыр фракциясының материалына зерттеу жүргізілді. Қождың орташа сынамасының рентгенодифрактометриялық талдауы cu-сәулеленуі бар ДРОН –4 дифрактометрінде, графитті монохроматорда орындалған. Сынамалардан ауыр фракциялар бөлініп, жылтыратылған жасанды аншлифтер (брикеттер) жасалды. Аншлифтер LEICA DM 2500p маркасының микроскопымен және иммерсиялық сұйықтықтармен зерттелді.

Зерттеу нәтижелері бойынша қорғасын қождарында түсті металдар қосылыстарының жоғары мөлшері бар екендігі анықталды: қорғасын оксиді 0,7%-ға дейін және мырыш оксиді қождың салмақтық мөлшерінің 8,5%-на дейін, бұл қорғасын өндірісінің улы қалдықтарын кәдеге жарату процесін техникалық және экономикалық тұрғыдан орынды етуге мүмкіндік береді.

Түйін сөздер: қалдықтар, қорғасын-мырыш қалдықтары, қайталама шикізат, түсті металдар, бағалы компоненттер, қалдықтарды қайта өңдеу, қалдықтарды кәдеге жарату, аз қалдықты және қалдықсыз технологиялар.

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АНАЛИЗ И ПЕРСПЕКТИВЫ УТИЛИЗАЦИИ ТЕХНОГЕННЫХ ШЛАКОВЫХ ОТХОДОВ СВИНЦОВОГО ПРОИЗВОДСТВА

Аннотация. В статье представлены анализ и результаты исследования техногенных шлаковых отходов свинцово-цинковых производств. Шлаки свинцово-цинковых производств содержат большое количество токсичных соединений: свинец, цинк, осмий, кадмий, которые являются опасными источниками экологического загрязнения. Из-за открытого хранения шлаков было выявлено превышение предельно допустимых концентраций свинца. Утилизация техногенных шлаковых отходов имеет большое значение для снижения отрицательного влияния на безопасность жизнедеятельности и улучшения экологической обстановки региона. В то же время шлаки являются ценным сырьем, содержащим соединения цветных и редкозе-мельных металлов.

В статье показаны результаты лабораторных исследований шлаков по определению качественного и количественного состава ценных компонентов в отходах свинцового производства и возможности дальнейшей их переработки и утилизации. Были проведены исследования материала тяжелой фракции шлака на электронно-зондовом микроанализаторе марки JEOL IXA–8230 Electron Probe microanalyzer. Рентгеноди-фрактометрический анализ средней пробы шлака выполнен на дифрактометре ДРОН-4 с Cu-излучением, графитовый монохроматор. Из проб были выделены тяжелые фракции и изготовлены полированные искусственные аншлифы (брикеты). Аншлифы изучались под микроскопом марки LEICA DM 2500P и в иммер-сионных жидкостях.

По результатам исследований выявлено, что в свинцовых шлаках содержится достаточно высокое количество соединений цветных металлов: оксида свинца до 0,7 % и оксида цинка до 8,5 % от весового количества шлака, что

позволяет сделать процесс утилизации токсичных отходов свинцового производства технически и экономически целесообразным.

Ключевые слова: техногенные отходы, свинцово-цинковое производство, вторичное сырье, цветные металлы, утилизация отходов производства, малоотходные и безотходные технологии.

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