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Satbayev University

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ИЗВЕСТИЯ

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РЕСПУБЛИКИ КАЗАХСТАН
Satbayev University

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Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар 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. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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DISPOSAL OF LEAD PRODUCTION WASTES BY EXTRACTION OF LEAD AND ZINC OXIDES

Abstract. Information is given about the need to dispose of waste from the Shymkent lead plant in the form of slags, which have accumulated about 2 million tons. It is proved that lead production slags contain a large number of toxic compounds, such as lead, zinc, osmium, and cadmium, which are dangerous sources of environmental pollution.

According to the results of X-ray diffractometric analysis and DTA, it was found that the slag of lead production contains a fairly 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 recycling toxic waste from lead production technically and economically feasible.

The results of preliminary tests allow us to select 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.

Keywords: waste, lead waste, toxic compounds, lead and zinc compounds, environmental pollution, toxic waste processing, waste disposal

Introduction. On the territory of South Kazakhstan region (now Turkestan region), in the city of Shymkent during the 30s of the last century, a lead plant for the production of lead operated. Even after its closure, the plant is environmentally unsafe. The technologies used at the plant were inferior in efficiency to modern analogues. The mined ore had a high lead concentration, which allowed the plant to neglect the residual metals in the slags due to their relatively low concentration. As a result of the activity of the enterprise, about 2 million tons of wastes in the form of lead slags have been accumulated [1-3]. Due to the open storage of slags, the environmental expertise revealed a huge excess of the maximum permissible concentration (MPC) of lead in the soil near the former lead plant: currently, the MPC near the plant is more than 3000 mg/kg in the soil, and according to the standards it should be 3.2 mg/kg. According to the analyzes carried out in all plant samples, the norm was exceeded from 1.83 to 8.13 times [4-5].

Lead production slags also contain a certain amount of potentially harmful elements, such as lead, zinc, osmium, cadmium, which are possible environmental pollution sources. The environmental damage of long-term storage of slag is seriously dangerous when the slag is in an acidic environment (soil pH < 4). Shymkent soil cover contamination with lead compounds of waste slags leads to the ingress of metal into the human body. Lead compounds negatively affect the human nervous system, which leads to a decrease in intellect, causes changes in physical activity, coordination of hearing, affects the cardiovascular system, leading to heart disease [6].

Lead occupies the first place among professional intoxications, and there is a tendency to its increase. Among workers affected by lead exposure, about 40% are women. Lead is particularly dangerous for women as this element has the ability to penetrate through the placenta and accumulate in breast milk. As a rule, the highest lead concentration in the atmospheric air is observed in winter, which is associated with additional emissions of fuel combustion products into the atmosphere. Unfavorable meteorological conditions during this period of the year also contribute to the lead accumulation in the lower atmosphere layers. Lead enters the body through the gastrointestinal tract or the respiratory system and is then carried by the blood throughout the body. Moreover, the inhalation of lead dust is much more dangerous than the presence of lead in food. Lead compounds also accumulate in bones, partially replacing calcium in phosphate. Entering in soft tissues – muscles, liver, kidneys, brain, lymph nodes, lead causes a disease – plumbism. Like many other heavy metals, lead (in the form of ions) blocks the activity of certain enzymes. The authors established that their activity decreases 100 times with an increase in the lead concentration in the blood 10 times – from 10 to 100 micrograms per 100 ml of blood. At the same time, anemia develops, the hematopoietic system, kidneys and brain are affected, and intellect decreases. This has a negative impact on the health of the population, and especially children, who are most susceptible to lead poisoning [7-8].

Lead can easily enter the body with drinking water if it comes into contact with metal: in the presence of carbon dioxide, soluble bicarbonate slowly passes into the solution. It is enough that there is only one milligram of lead in a liter of water – and drinking such water becomes very dangerous. The lead accumulation is intensively carried out by fungi, mosses and lichens and bring its concentration to 64.76 ppm, respectively. The more familiar oats and clover, already at a lead concentration of 50 ppm, begin to slow down growth and yield declines. Lead comes from the atmosphere into the soil most often in the form of oxides, where it gradually dissolves, turning into hydroxides, carbonates or the form of cations [9]. The main source from which lead enters the human body is food, along with this, inhaled air plays an important role, and in children also the lead dust they ingest. Inhaled dust is retained in the lungs by about 30-50%, its significant part is absorbed by the blood stream. Absorption in the gastrointestinal tract is generally 5-10%, in children – 50%. Calcium and vitamin D deficiency enhances the lead absorption in the gastrointestinal tract. On average, the human body absorbs 26-42 μg of lead per day. This ratio may vary. About 90% of the total amount of lead in the human body is found in bones, in children – 60-70%. In addition to lead, lead production slags contain zinc compounds, which also adversely affect the environment: wastewater containing zinc is not suitable for irrigation of fields, and the negative zinc effect on microorganisms and soil microfauna significantly reduces soil fertility. Many manifestations of zinc intoxication are based on the competitive relationship between zinc and a number of other metals. Residents of nearby areas showed a significant decrease in total serum calcium levels [10-12].

Excessive zinc ingress into the body of animals was accompanied by a drop in the calcium content not only in the blood, but also in the bones, while the absorption of phosphorus was disrupted; as a result, osteoporosis developed. The toxicity of zinc oxide is explained by its catalytic activity. Zinc can be mutagenic and oncogenic. Thus, due to the great harm to the health of the population of neighboring regions, the problem of lead production slags disposal is urgent [13-15].

At the same time, the lead plant slags are an important raw material containing various non-ferrous metals, and at present 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 volume of non-ferrous metals is about 25%. For example, lead has a high economic value and the applications of lead have changed in recent years, and now approximately 80% of world consumption fall at the electric battery production sector. The flexibility, density and anti-corrosion properties of lead are still actively used in the construction of tanks for storage of caustic liquors and as protection against X-rays and radiation. Lead is used in the manufacture of paints and pigments and other chemical compounds [16].

Other major consumer of lead is 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 included in bronzes, brasses, babbits, printing alloys [17-18].

Zinc is another non-ferrous metal offered for extraction in the lead production slag disposal process. Zinc is also used for zincing metal products in order to give them anti-corrosion properties. Zinc is widely used in the production of alloys (brass, cupronickel), printing materials, rolled products and zinc oxide [19]. The demand for zinc remains strong due to the explosive growth in the production of anti-corrosion coatings. Zinc compounds are also used in the manufacture of pigments for paints, rubber, glass and glazes. Another important area of application is in neutralizing cosmetic pastes and pharmaceuticals. After extraction of non-ferrous metals from slags, they can be used in the production of cement, building materials, since the lead production slags contain FeO, CaO, SiO_2 in an amount of about 75-85% [20-21].

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 [2]. 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 analyzes. 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 = 35 kV; I = 20 mA; 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.

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. The identification of mineral phases according to the data of X-ray diffractometric analysis is shown in the diffractogram in figure 1.

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

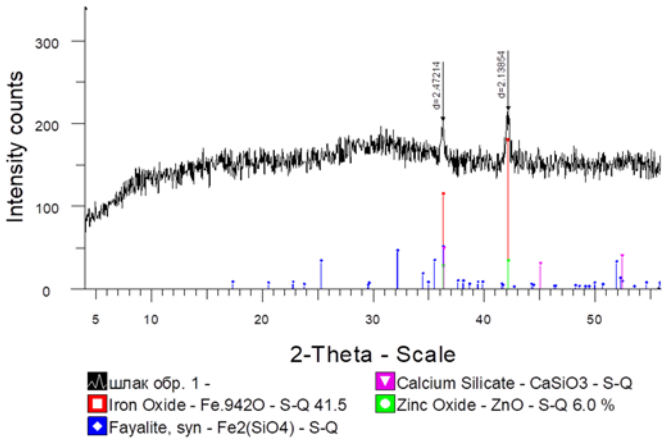

Figure 1 –
Diffractogram of the slag sample

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.

Thermal analysis of the slag samples was carried out according to DTA and TGA measurements. The thermal analysis of the powder slag sample was carried out on Q-1000/D derivatograph of F. Paulik, J. Paulik and L. Erdey systems of "MOM" company (Hungary, Budapest). The survey was carried out in the air, in the temperature range 20-1000°C, heating mode – dynamic (dT/dt = 10), reference substance – calcined Al_2O_3, sample weight – 500 mg with a valuable division of the sample weight change scale – 500 μV. The research revealed the following parameters: the sensitivity of the balance is 100 mg, the sensitivity of other measuring systems of the device: DTA = 250 μV, DTG = 500 μV, TG = 500 μV T = 500 μV.

The method used is based on the device registering changes in the thermochemical and physical parameters of a substance, which can be caused during its heating. The thermochemical state of the sample is described by the curves: T (temperature), DTA (differential thermoanalytical), TG (thermogravimetric) and DTG (differential thermogravimetric), the last curve is a derivative of the TG-function.

The optimal thermochemical parameters obtained during high-temperature processing of the test system allowed to reveal the nature of the destruction of thermally active components.

The powder sample composition was identified by the thermal curves morphologies and the obtained numerical values of the intensities of endo- and exothermic effects, using the thermogravimetric readings of the TG lines coupled with them [22].

The results of the analysis were compared with the data given in the atlases of the thermal curves of minerals and rocks and compared with the descriptions of the thermal behavior of monomineral samples set forth in other reference sources and accumulated in the data bank of the laboratory conducting these studies.

The slag sample in dynamic heating mode on (DTA-, DTG- and TG-) curves in different temperature ranges left a series of effects caused by endo- and exothermic reactions, as shown in figure 2 and their quantitative values in table 5.

Within the limits of low temperatures (20-200°C) in the system under study, the endothermic effect was noted with the weight loss Δm1 equal to 1.75% of the sample mass shown in figure 2, table 5. Many powder samples contain atmospheric water, which in the specified temperature range is carried out into the atmosphere. In this case, the main part of the evaporated molecular water can be attributed to the dehydration of particles of the powder slag adsorbed H_2O. After the sample dehydration process, the enthalpy of the system in the range of 200-280°C practically does not change, which is caused by the absence of weight loss in this temperature range. It should be noted that in the range of 280-930°C the thermogravimetric curve (TG) is steadily shifting upward – towards an increase in the sample mass, which is caused by the introduction of atmospheric oxygen into the system. The increase in mass is accompanied

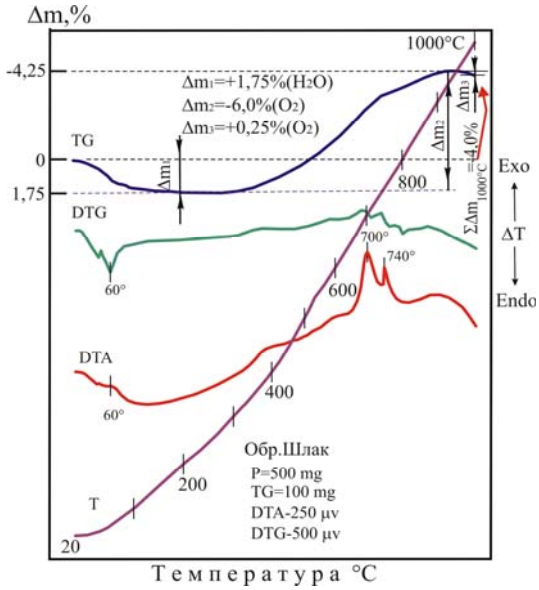

Figure 2 –
Derivatogram of the slag sample

Table 5 – The sequence of the quantitative values of the weight loss of volatile components

Sequence of the weight loss	Weight loss, %	Volatile components	Decomposition temperature range, °C
Δm_1	1.75	H_2O	20-200
Δm_2	-6.0	$-O_2$	280-930
Δm_3	0.25	O_2	930-1000
$\Delta m_{1000°C}$	4.0	H_2O, $-O_2$, O_2	20-1000
Note to figure 2 and to table 5: The minus sign (-) at the Δm2 value means the introduction of atmospheric oxygen into the system. This sign is the opposite of the sign applied to the weight loss parameter.			

by a rise in the DTA-curve line, which, in the range of 640-800°C, formed distinct exothermic peaks at 700 and 740°C. The processes that caused the introduction of heat into the system are associated with the oxidation of the ferrous components of the sample. Oxygen-enriched iron oxides are usually formed within the indicated temperature ranges. These high temperature peaks are caused by the transition of iron oxide from a lower acidity level to a higher level. At a higher temperature (930°C), the increase in sample mass reaches its limit (Δm2 = -6%). And further heating of the sample (up to 1000°C) leads to a decrease in its mass by 0.25% [23].

According to the results of the X-ray diffractometric analysis of the slag samples, the formation of the following useful components was revealed: copper in the form of sulfides, complex compounds of oxides of lead, zinc, iron and copper, which are in the form of inclusions in an amorphous host matrix of complex composition. The results of the laboratory studies of the lead production slag showed a sufficiently high content of non-ferrous metal compounds, which allows to evaluate the process technically and economically efficient when extracting them during the lead production toxic wastes' disposal.

Conclusion. Modern methods of analysis were used to determine the chemical composition of waste from the Shymkent lead plant (JSC Yuzhpolimetal). The results of X-ray diffractometric analysis and DTA showed that the slag of lead production contains a fairly high amount 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.

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 of 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 waste.

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ҚОРҒАСЫН ЖӘНЕ МЫРЫШ ОКСИДТЕРІН АЛУ АРҚЫЛЫ ҚОРҒАСЫН ӨНДІРІСІНІҢ ҚАЛДЫҚТАРЫН КӘДЕГЕ ЖАРАТУ

Аннотация. Шымкент қорғасын зауытының қалдықтарын 2 млн тоннаға жуық қож түрінде кәдеге жарату қажеттілігі туралы мәліметтер келтірілді. Қорғасын өндірісінің шлактарында экологиялық ластанудың қауіпті көзі болып табылатын қорғасын, мырыш, осмий, кадмий сияқты көптеген улы қосылыстар бар екендігі дәлелденді.

Рентгенодифрактометриялық талдау және ДТА нәтижелері бойынша қорғасын өндірісінің қожында түсті металдар қосылыстарының жеткілікті жоғары мөлшері бар екендігі анықталды: қорғасын оксидінің мөлшері 2%-ға дейін, мырыш оксиді 17%-ға дейін және мыс оксиді сынаманың жалпы салмағының 1,25%-на дейін. Қорғасын қождарының түсті металдардың сапалық құрамы мен қорғасын өндірісінің улы қалдықтарын кәдеге жарату процесін техникалық және экономикалық тұрғыдан орынды етуге мүмкіндік береді.

Алдын ала сынақтардың нәтижелері қорғасын өндірісінің қожды қалдықтарынан қорғасын және мырыш оксидтерін неғұрлым толық және селективті алу үшін технологияны таңдауға мүмкіндік береді. Түсті металдарды алудың селективті тәсілін қолданған кезде қорғасын өндірісінің уытты шлактарын кәдеге жарату есебінен қоршаған ортаның экологиялық жай-күйін жақсарту және адамдардың денсаулығына теріс әсерді азайту күтіледі. Бұл ретте табиғи және қайталама ресурстарды ұтымды пайдалану жүйесін дамытуға елеулі үлес қосылады.

Түйін сөздер: қалдықтар, қорғасын қалдықтары, улы қосылыстар, қорғасын және мырыш қосылыстары, экологиялық ластану, улы қалдықтарды қайта өңдеу, қалдықтарды кәдеге жарату.

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УТИЛИЗАЦИЯ ОТХОДОВ СВИНЦОВОГО ПРОИЗВОДСТВА ПУТЕМ ИЗВЛЕЧЕНИЯ ОКСИДОВ СВИНЦА И ЦИНКА

Аннотация. Приведены сведения о необходимости утилизации отходов Шымкентского свинцового завода в виде шлаков, которых накопилось около 2 млн тонн. Доказано, что шлаки свинцового производства содержат большое количество токсичных соединений, таких как свинец, цинк, осмий, кадмий, которые являются опасными источниками экологического загрязнения.

По результатам рентгенодифрактометрического анализа и ДТА выявлено, что в шлаке свинцового производства содержится достаточно высокое количество соединений цветных металлов: содержание оксида свинца до 2 %, оксида цинка до 17 % и оксида меди до 1,25 % от общего веса пробы. Качественный состав и содержание цветных металлов свинцовых шлаков позволяет сделать процесс утилизации токсичных отходов свинцового производства технически и экономически целесообразным.

Результаты предварительных испытаний позволяют выбрать технологию для более полного и селективного извлечения оксидов свинца и цинка из шлаковых отходов свинцового производства. При применении селективного способа извлечения цветных металлов ожидается улучшение экологического состояния окружающей среды и уменьшение отрицательного влияния на здоровье людей за счет утилизации токсичных шлаков свинцового производства. При этом вносится весомый вклад в развитие системы рационального использования природных и вторичных ресурсов.

Ключевые слова: отходы, отходы свинца, токсичные соединения, соединения свинца и цинка, экологическое загрязнение, переработка токсичных отходов, утилизация отходов.

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