MODERN METHODS FOR PROCESSING IRON-CONTAINING TECHNOGENIC WASTE (TAILS) OF COPPER PRODUCTION

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Abstract. *Currently, the depletion of mineral resources and the continuous increase in their production capacity of processing enterprises and the demand for commercial products leads to increasing volumes of pollution (tailings, sludge, slag, etc.) of the natural environment and environmental requirements are increasingly involved in circulation secondary techno genic sources of minerals.*

Iron is the most important element for the development of human civilization. Iron-based materials are used in almost all areas of industry.

The largest volume of iron-containing waste is tailings from concentration factories, which contain large amounts of magnetite, hematite, fayalite, bornite, tetrahedrite from copper concentration factories.

Currently existing methods and technological schemes for processing iron-containing tailings and waste are faced with the problem of low economic efficiency, the degree of extraction of iron and other valuable associated elements.

Keywords*: tailings, sludge, slag, magnetite, Fe_3O_4, enrichment, flotation, iron-containing waste.*

Introduction. The material composition of enrichment waste, as well as processing technologies, differ significantly from the composition of the original raw materials and technological enrichment schemes. Concentration tailings contain from 25 to 45% Fe. In this case, iron is found in the form of weakly magnetic hematite and other non-magnetic mineral compounds. To achieve high efficiency in the processing of iron-containing waste in order to obtain valuable components, it is necessary to apply technologies of high-intensity magnetic separation, cationic flotation, as well as different modes of thermal treatment of feedstock. [1,2].

Iron-containing waste is formed depending on the conditions of deposit formation, mining methods, enrichment technology and metallurgical processing. The iron content in industrial ores varies widely from 16 to 70%. The most important ore minerals are: magnetite, magnetite, titanomagnetite, hematite, siderite (iron carbonate), ferruginous chlorites (chamosite, thuringite, etc.). In this regard, waste from mining and metallurgical enterprises is being actively studied in order to determine the possibility of producing high-quality iron ore raw materials from them. [3]. Waste from mining and metallurgical production contains a significant amount of non-ferrous, ferrous, noble, rare and trace metals and represents techno genic raw materials that were stored and accumulated due to the lack of economical and environmentally friendly technologies for their processing and disposal.

It should be noted that waste, being a powerful source of environmental pollution, is a valuable raw material for industry. Reducing the harmful impact of mining on the environment can be achieved by improving technology [4, 5].

Waste-free and low-waste technology is a modern direction for the development of industrial production. The main goal of this direction is to reduce the harmful effects of industrial waste on the environment. Non-waste technologies provide the opportunity for complex

processing of raw materials, and the development of such technological processes helps. As a result of the development of these technologies, it is possible to use natural resources more efficiently, recycle industrial waste, reduce the amount of waste and reduce its negative impact on the environment. [5]

Currently, the share of recycling waste from mining and processing industries, even in technologically developed countries, does not exceed 10% [6].

The lack of coordination in the extraction of individual components leads to the fact that when only a few components are extracted from complex raw materials, other, no less valuable, but non-recoverable components with high content end up in waste. The least developed and therefore most dangerous problem remains the processing of mineral masses deposited on the earth's surface. The constant lag between processing capabilities and production capabilities increases the relevance of this problem, making it global and fundamental for humanity [7].

Tailings from the enrichment of pyrite ores and pyrite products constitute one of the dangerous groups of techno genic waste and at the same time are an accumulator of heavy, non-ferrous and noble metals, as well as rare elements. Up to 35-40% of the valuable components in these products are in aggregates, the other part is represented by free particles with a particle size of 10 microns. Unraveling thin aggregates of ore and rock minerals is an extremely difficult task, even with modern ultrafine grinding capabilities. The material composition of sulfide minerals in the process of ore beneficiation changes with the formation of new mineral phases, the extraction of which is possible only by chemical-metallurgical methods or using energy influences [8,9].

Theoretical foundations of reduction firing. The purpose of reduction roasting is to convert metal oxides contained in wastes and concentrates into the metallic state or into lower oxides. It is known that reduction processes are widespread in metallurgy, where they have independent significance as methods for producing finished metals from their oxides. We will limit ourselves only to the reduction roasting of various ore materials, as a preparatory operation for their enrichment and metallurgical processing. An example of such operations is magnetizing roasting of oxidized iron compounds in order to convert iron oxides into highly magnetic ones (mainly magnetite) with their subsequent concentration on magnetic separators. Magnetizing roasting is also used for preparation for the enrichment of nickel-containing laterite and titanium-zirconium-containing ores, for deferrization of high-iron bauxites and in other processes.

The reduction process also produces metal products from high purity concentrates, which are the starting materials for the production of iron, nickel, copper and other metal powders.

A similar process, but with reduction not to magnetite, but to metal; is metallizing roasting of iron ores. In some cases, this makes it possible to obtain a significant technical and economic effect in metallurgical production. For poor, difficult-to-process iron ores, sometimes metallizing roasting is a preparatory operation before beneficiation. The fired product with inclusions of metallic iron particles, after grinding to the appropriate size to ensure their opening, is subjected to magnetic separation. The resulting iron-containing concentrates are used as metal charge in steel-smelting furnaces. [8].

Technology of magnetizing roasting of iron-containing material Magnetizing roasting is heating and holding iron ores in a reducing gas environment (with a lack of oxygen) to convert non-magnetic iron oxides mainly α-Fe_2O_3 (hematite) into magnetic ones-Fe_3O_4 (magnetite) and γ-Fe_2O_3 (maghemite). During magnetizing firing, hematite is reduced to magnetite:

$$3Fe_2O_3 + CO \rightarrow 2Fe_3O_4 + CO_2, -37,25 \text{ MJ} \quad (1)$$
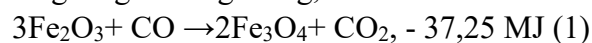
The ore is then cooled in an environment with excess CO_2; in this case, particles reduced to FeO (wustite) are oxidized to Fe_3O_4:

$$3FeO + CO_2 \rightarrow Fe_3O_4 + CO, \quad (2)$$
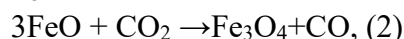
When the reduced material is cooled to a temperature at which magnetite is stable (300-400 °C), partial oxidation of Fe_3O_4 to highly magnetic γ-Fe_2O_3 (maggetite) occurs. After magnetizing roasting, the ore is subjected to magnetic separation in a weak magnetic field (H=90-140 кА/м).

Magnetizing firing technology is implemented in several furnace units. Shaft kilns have gained some popularity in KHP for firing hematite quartzites, as well as for firing siderites. Furnaces in China are narrow (from 900 to 1800 mm) chambers, gradually widening towards the bottom. In the middle part they have a constriction, dividing them into two zones: the upper heating zone and the lower recovery zone. Adjacent to the longitudinal walls are two chambers for pre-combustion of coke oven gas. The products of its combustion with a temperature of about 1270 °K enter through channels into the middle part of the furnace and, rising towards the descending ore, heat it to 870-970 °K. Reducing gas is supplied to the lower part of the furnace through a distribution channel. The need to burn fuel in the side chambers is due to the desire to eliminate the fusion that occurs when burning gas or pulverized fuel in the ore layer. Melting is associated with a high concentration of combustion on the surface of the ore pieces due to its surface (catalytic) nature. And then the ore is cooled in an environment with excess CO_2; in this case, particles reduced to FeO (wustite) are oxidized to Fe_3O_4:

$$3Fe_2O_3 + H_2= 2Fe_3O_4+ H_2O + 4,2 \text{ МДж (3)}$$
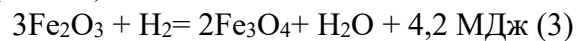
After magnetizing roasting, the ore is subjected to magnetic separation.

At temperatures below 845 °K, thermodynamic reduction should occur without the formation of wustite.

In real conditions, the contact of solid phases of iron oxides and carbon is limited; reduction processes with solid carbon should also be considered taking into account their flow through the gas phase according to the previously described reactions (1). In this case, the formation of reducing gases occurs through the reactions: $CO_2+C=2CO$; $H_2O+C =H_2+CO$. The gasification reaction of carbon with its dioxide (Bell-Boudoir reaction) depends on pressure and is reversible.

Materials are unloaded from the furnace from both sides using drum feeders. To prevent air from entering the reduction zone through the discharge openings, water seals are used. The roasted material is discharged into water and onto plate conveyors, which feed the ore into the hoppers of the crushing department, where it is crushed and fed to wet magnetic separation. With a crude ore content of 33% Fe, concentrate 11 contains 56% Fe, and tailings contain 14.6% Fe. The recovery of iron into the concentrate was 76%.

The productivity of furnaces of this type is 5-10 t/m^3 of internal volume per day, but their structural complexity does not allow the creation of shaft furnaces of large unit capacity (above 1000 t/day). Therefore, despite all their advantages, they are not universal and can only be recommended in combination with other types of furnaces for roasting large classes of ore to be processed [9].

Tubular rotary kilns are the most widely used in the practice of magnetizing roasting of ores. Such a stove has a slope to the horizon of up to 5 °C. Its upper end is closed by the loading head, and the lower end by the unloading head. It works on the counter flow principle, i.e. the loaded materials, forming an overflowing layer, move towards the hot gases washing them.

The material is reduced both by brown coal supplied in a mixture with it, and by natural gas blown through a layer of heated material at the discharge end of the furnace. Intensification of the thermal regime of the furnaces can be achieved by installing an additional central gas or pulverized coal burner at the discharge end. This will increase the ore recovery rate from 70-80% to optimal and improve enrichment performance.

A solid reducing agent, compared to a gaseous one, significantly improves one of the serious disadvantages of the rotary kiln, which is the relatively small contact surface of the ore with the reducing gases. Heated to 1070-1120 °K and partially reduced ore in a mixture with a solid reducing agent enters the post-reduction chamber, where the conversion of Fe oxides into magnetite is completed. The oven operates on the counter flow principle. For magnetizing roasting of iron ores, other conditions are needed, namely, the supply of large amounts of heat to the layer for heating the material and the creation of a reducing gas environment.

Currently, two types of fluidized bed furnaces have been tested for magnetizing roasting of iron ores: single-zone and multi-zone. In a single-zone furnace, the roasting zone combines the heating of ore and the reduction of Fe oxides to magnetite. The heat from the gases leaving the firing zone is transferred to an additional drying unit attached to the fluidized bed furnace. The disadvantages of the process are the consumption of expensive coke reducing agent, the duration of the roasting-reduction process, incomplete recovery of pyrite minerals, and incomplete extraction of valuable components from the processed material.

OJSC "Uralmekhanobr" has developed a combined flotation-hydrometallurgical technology for the enrichment of techno genic raw materials with a high content of copper sulfate. The enrichment technology has been developed for raw materials with different contents of copper sulfate: - for fine copper ore containing 57.9% (rel.) of water-soluble sulfate - chalcanthite; - for old pyrite tailings of the Buribayevsky GOK with a content of water-soluble copper sulfates (chalcanthite) equal to 26.1% (rel.). The combined technology involves initial leaching with the release of water-soluble copper into the productive solution [10]. The washed ore is then subjected to flotation enrichment. Copper compounds were isolated from an aqueous solution using chemical precipitation or cementation methods. Chemical precipitation was carried out with sodium sulfide, cementation with iron powder.

To precipitate copper sulfide from the Blavinsky tailings (fine copper ore), a saturated solution of sodium sulfide Na_2S was initially used. It was noted that during water washing, together with copper sulfate, part of the iron (II) and iron (III) passes into the aqueous phase in the form of sulfates [6].

The proposed deposition process occurs according to the reactions [11]:

$$Cu^{2+} + S^{2-} = CuS\downarrow \quad (1)$$
$$2Fe^{3+} + S^{2-} = 2Fe^{2+} + S°\downarrow \quad (2)$$
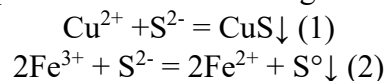
The optimal consumption of sodium sulfide was 2.95 kg/kg of copper in solution. As a result of precipitation, a precipitate with a copper content of 27.8% is obtained, with its extraction from the solution being 99.8%. The high consumption of the precipitant is explained by the presence of large quantities of iron (III) in the solution, as a result of which the main part of sodium sulfide is spent on a side reaction (2). The possibility of copper precipitation with a cheaper reagent was also studied - a saturated solution of sodium hydrosulfide NaHS, the effect of which is similar to sodium sulfide. The optimal consumption of sodium hydrosulfide was 7.1 kg/kg copper.

The disadvantage of the process is that copper sulfide technology is used; the processed material also contains a significant amount of iron sulfides, which will precipitate along with copper sulfides. The extraction of other valuable components from the feedstock is not considered..

1.3. Opening of iron-containing waste with ammonium chloride

Currently, many physical methods of ore beneficiation are unprofitable. Some types of ores cannot be enriched at all, which is why they are taken out of use. For example, most of the nickel is found in oxidized nickel ores, but its content in the ore is no more than 1%. To extract nickel, the entire ore mass must be smelted into matte. This leads to enormous energy costs and the unprofitability of the entire technology as a whole. [12].

The chemical composition of the dump slag from the processing of copper ores of Almalyk MMC JSC is given in the table 1.

Table 1.

Chemical composition of copper ore processing waste slag
JSC "Almalyk MMC"

Name	Quantity	Chemical components, %								
		Cu	Pb	Zn	S	Au	Ag	Fe	Mo	SiO$_2$
Reflective and oxygen-torch smelting slags	14 th-d. t.	0,6-0,8	0,1	0,9	1,2	$4*10^{-5}$	$17*10^{-5}$	35	0,03	33-39

To replace the existing physical methods of enrichment, the Fluoride Technologies company proposes to consider a fundamentally new approach to the practice of enrichment - using dry enrichment with a regenerated agent.

It is proposed to use ammonium halides – ammonium fluoride or ammonium chloride – as the main agent. The uniqueness of these compounds lies in the fact that they are able to bind a valuable component of the ore mass into a gaseous or soluble compound and separate it from the total mass of ore.

For the first time, it became possible to introduce into industrial use mineral raw materials that were not subject to enrichment. Technologies for ammonium halogen processing of techno genic mineral raw materials have been developed. Currently, feasibility studies and regulations have been prepared for the halo ammonium processing of a number of techno genic wastes and enriched concentrates. In particular, processing slag from copper smelters; processing of tailings from hydrometallurgical plants; processing waste from gold recovery factories.

The principle of ammonium halide separation of oxide mineral raw materials into individual oxides has been developed. In depth studies of the physical and chemical foundations of halo ammonium technology processes are accompanied by laboratory testing of each method on real man-made waste and end with a feasibility study and comparison of the economic indicators of existing technologies with developed ones. It should be noted that the use of the ammonium halide method eliminates the formation of slag and sludge - all ore raw materials are divided into commodity options, which are further involved in the commercialization process. The absence of waste also has a positive effect on the economics of the process in terms of ecology and the cost of disposal of sludge, slag, "tailings", etc. in classical methods. The use of the proposed methods seems especially beneficial in the modern economic situation, when the cost of the main reagent of hydrometallurgy - sulfuric acid - reaches 200 dollars/t. In this case, sulfuric acid is used only once and cannot be regenerated. The main reagent of halo ammonium technology, ammonium fluoride, can be used repeatedly. At the same time, the cost of its regeneration is no more than 30 dollars/t. [12].

Currently, pilot testing of ammonium fluoride technology for the production of valuable components and pure technical silicon is planned.

There are a number of monographs [2] on the scientific foundations of ore beneficiation technology, hydrometallurgical and pyro metallurgical methods of ore processing and the integrated use of raw materials.

According to experts, the practical implementation of already developed technical solutions for the development of techno genic deposits will reduce the volume of mineral production by 20-30%.

The low-waste (non-waste) technology considered by the authors can be developed in the following directions: creation of drain less technological systems based on existing, implemented and promising treatment methods;

- development and implementation of systems for processing secondary raw materials;

- creation of territorial-industrial complexes (TIC) with a closed structure of material flows of raw materials and waste within the TIC, with a minimum of emissions. The process takes place in three stages:

1. Dissolving iron with an aqueous solution of sulfuric acid, the concentration of iron sulfate is about 120 g/l;

2. Hydrate oxidation:

3. Pigment synthesis.

After oxidation, hydrates precipitate. Ammonia is used for precipitation. The process is carried out at room temperature, according to the reactions (1), (2).

$$2FeSO_4 + H_2O + 1/2O_2 = 2Fe(OH)SO_4 \ (1)$$
$$Fe(OH)SO_4 + 2NH_3 + 2H_2O = FeO(OH) + (NH_4)_2SO_4 + H_2O \ (2)$$
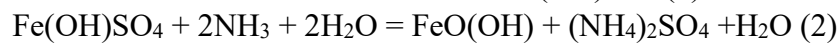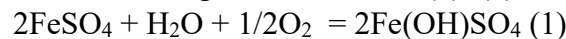
Seed crystals are single crystals of $FeO(OH)$, having sizes of 40-150 angstroms, depending on the synthesis conditions. When obtaining nuclei, the acidity of the solution increases slightly due to the hydrolysis reaction of the iron (II) salt in the solution (3):

$$Fe(OH)SO_4 + 2H_2O = Fe(OH)_3 + H_2SO_4 \ (3)$$
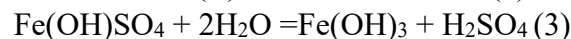
The embryos prepared in the reactor are poured into a container, from which they are fed using a pump into the reactor for pigment synthesis in an amount of 10-15% by weight of the pigment.

Pigment synthesis is carried out at a temperature of 60-70°C by oxidation of an aqueous solution of ferrous sulfate (concentration 120 g/l) with air oxygen.

During the synthesis, the pH of the reaction mixture is adjusted from 5.2 to 2.9-3.0, adjusting the pH with the amount of air supplied to oxidize iron compounds.

After the synthesis of the pigment is completed, the material is subjected to filtration to separate the pigment from the mother liquor into a suspension on a drum vacuum filter.

The washed sediment is usually dried in a continuous turbine-shelf dryer to obtain yellow pigments - $FeO(OH)$ and by calcination in a drum rotary kiln to obtain red pigments - Fe_2O_3.

The quality of the resulting pigment, especially color, dispersion and microscopicity, depends on the temperature of drying and calcination of the pastes. To obtain a yellow pigment, the drying temperature is 120°C, to obtain a red pigment - 750-800°C at the inlet and 250-300°C at the outlet. The flue gases leaving the dryer and puncture furnace contain up to 5% pigment, which are captured using electrostatic precipitators.

Results and discussion. A characteristic feature of the raw material from iron-containing tailings of processing plant-2 of Almalyk MMC JSC is its easy availability. All flotation tailings are located on the surface, lie compactly and, accordingly, do not require large extraction costs, i.e. the labor-intensive and expensive process of extracting rocks from a monolithic massif (drilling and blasting holes, loading and blasting, rock excavation) is eliminated. A number of processes occurring during the formation of tailings dumps lead to the concentration of residual metal contents in them, and the process of studying it is labor-intensive [13]

The absence of pyrite flotation from the current flotation tailings of the processing plant-2 of Almalyk MMC JSC after drying and storage is due to the screening of the surface by calcium carbonate, formed after the suppression of pyrite with lime. [14]

The chemical and elemental composition of iron-containing waste MOF-2 was carried out in a laboratory analyzer Rigaku XRF (Japan) X-ray fluorescence spectrometer. The mineralogical composition of iron-containing waste was determined using a diffractometric analyzer.

Analyzed result(FP method)

| No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Mg	0.907	mass%	0.0203	0.0351	0.105
2	Al	5.11	mass%	0.0195	0.0116	0.0349
3	Si	22.9	mass%	0.0224	0.0014	0.0041
4	S	0.802	mass%	0.0028	0.0042	0.0127
5	K	2.80	mass%	0.0273	0.0204	0.0612
6	Ca	2.59	mass%	0.0217	0.0185	0.0555
7	Ti	0.286	mass%	0.0059	0.0110	0.0331
8	Cr	0.0421	mass%	0.0017	0.0039	0.0117
9	Mn	0.323	mass%	0.0043	0.0057	0.0172
10	Fe	58.8	mass%	0.0366	0.0041	0.0123
11	Co	0.174	mass%	0.0072	0.0212	0.0637
12	Cu	0.931	mass%	0.0057	0.0024	0.0071
13	Zn	2.39	mass%	0.0076	0.0015	0.0046
14	As	0.0290	mass%	0.0020	0.0056	0.0167
15	Rb	0.0215	mass%	0.0005	0.0011	0.0033
16	Sr	0.0295	mass%	0.0004	0.0003	0.0009
17	Y	0.0069	mass%	0.0003	0.0009	0.0027
18	Zr	0.380	mass%	0.0048	0.0017	0.0052
19	Mo	0.299	mass%	0.0093	0.0057	0.0170
20	Ag	0.0025	mass%	0.0005	0.0008	0.0025
21	Sn	0.0121	mass%	0.0008	0.0012	0.0037
22	Sb	0.0869	mass%	0.0020	0.0014	0.0043
23	Ba	0.248	mass%	0.0041	0.0053	0.0159
24	Ir	(0.0112)	mass%	0.0020	0.0057	0.0170
25	Pb	0.554	mass%	0.0040	0.0038	0.0113
26	Ac	0.0336	mass%	0.0028	0.0081	0.0243
27	U	(0.0020)	mass%	0.0007	0.0020	0.0059

Spectrum
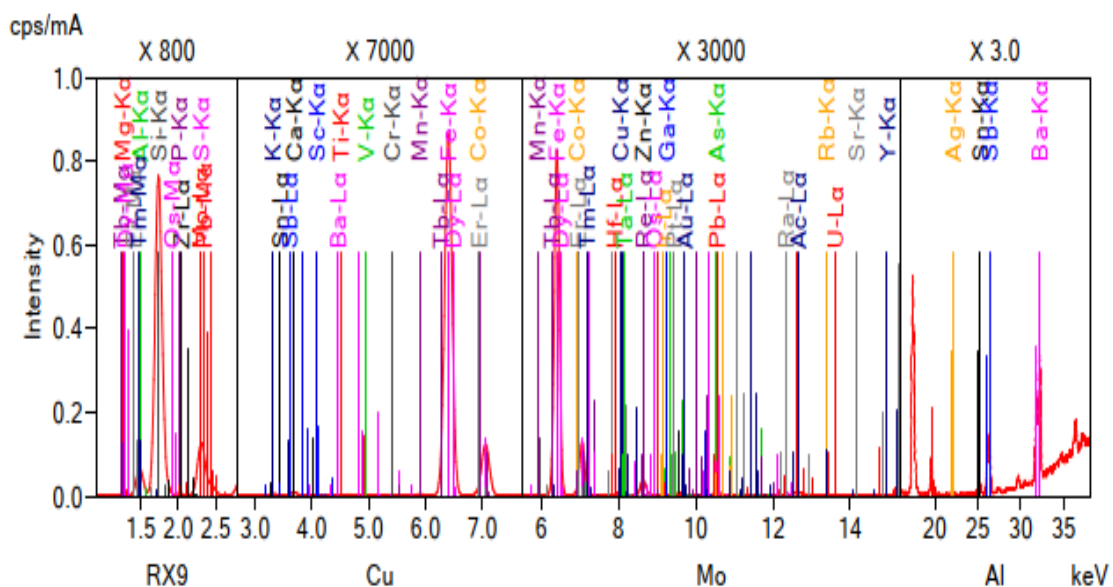
Pic. 1. Spectrum of elemental composition of iron-containing tailings

Main indicators of the device:

Diffraction patterns were recorded on an Empyrean device (Malvern Panalytical), Cu K radiation, Ni filter, with a graphite monochromator ($\lambda = 1.54178$ Å). Tube mode (Cu) 10 mA, 30 kV. The range of angle 2θ values is from 20 to 90°, step 0.013°, slit 0.6 mm, exposure at a point – 1 sec, energy discriminator – 0.17-0.23 keV. The spectrum was interpreted and the phase composition was calculated using the JCPDS-ICDD library using specialized software.
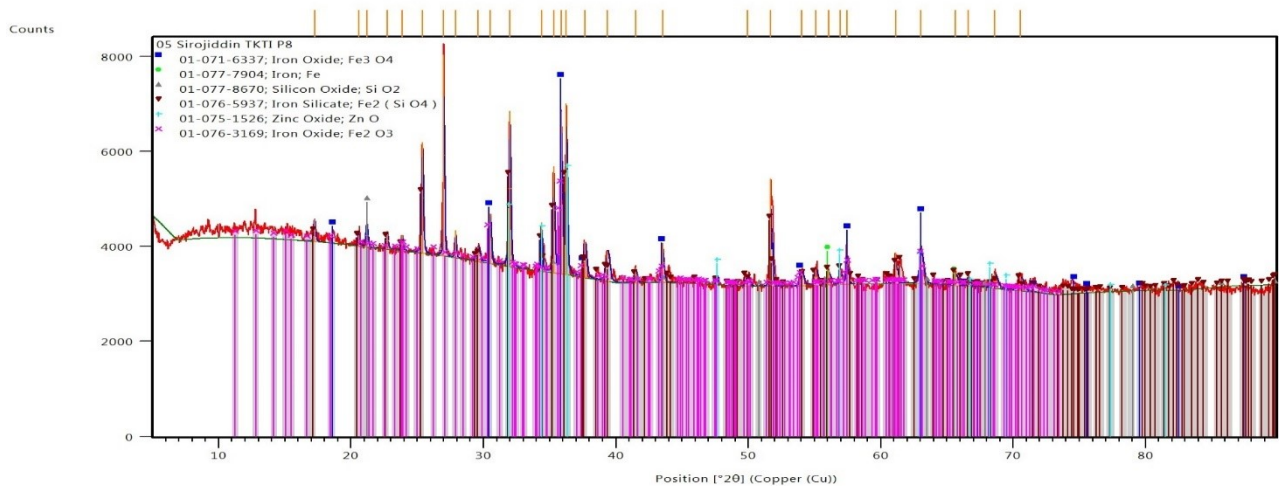
Pic. 2. Diffractometric analysis results of iron-containing tail

From Fig. 2 it is clear that in the range of peaks 10-20, 30-40, 40-50, 50-60, 60-70, 70-80, 80-90 the spectra of magnetite were reflected, and in peaks 30-40 the magnetite indicators are higher , fayalite spectra were reflected in the peak range 20-90.
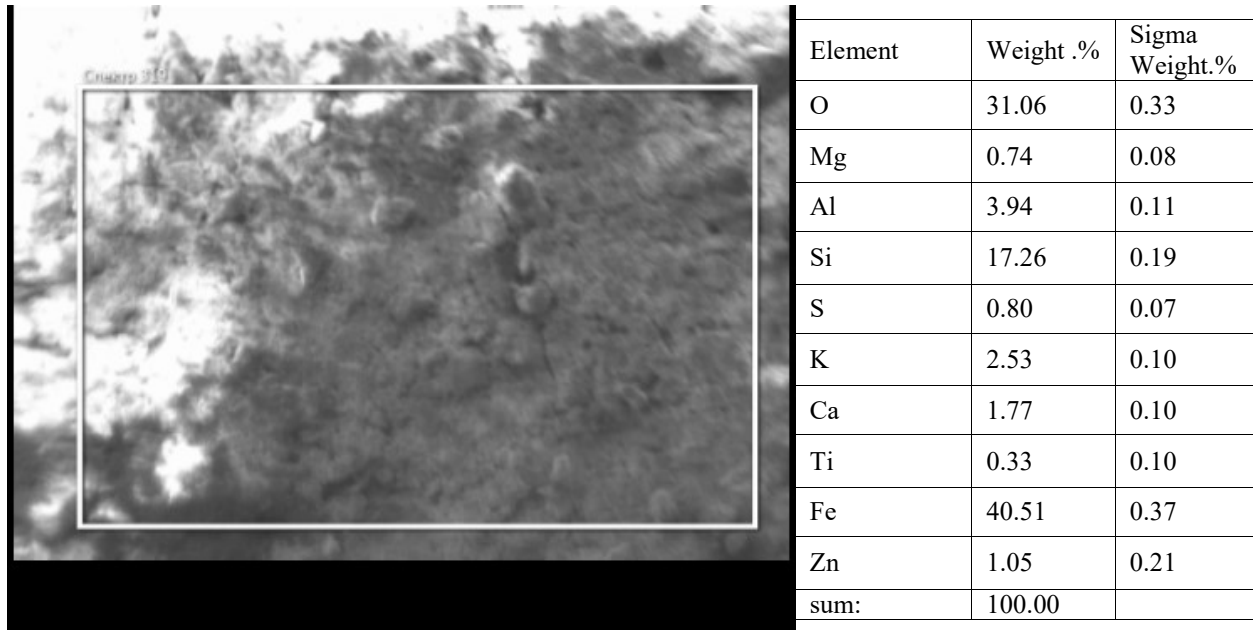
Indicators of diffractometric analysis
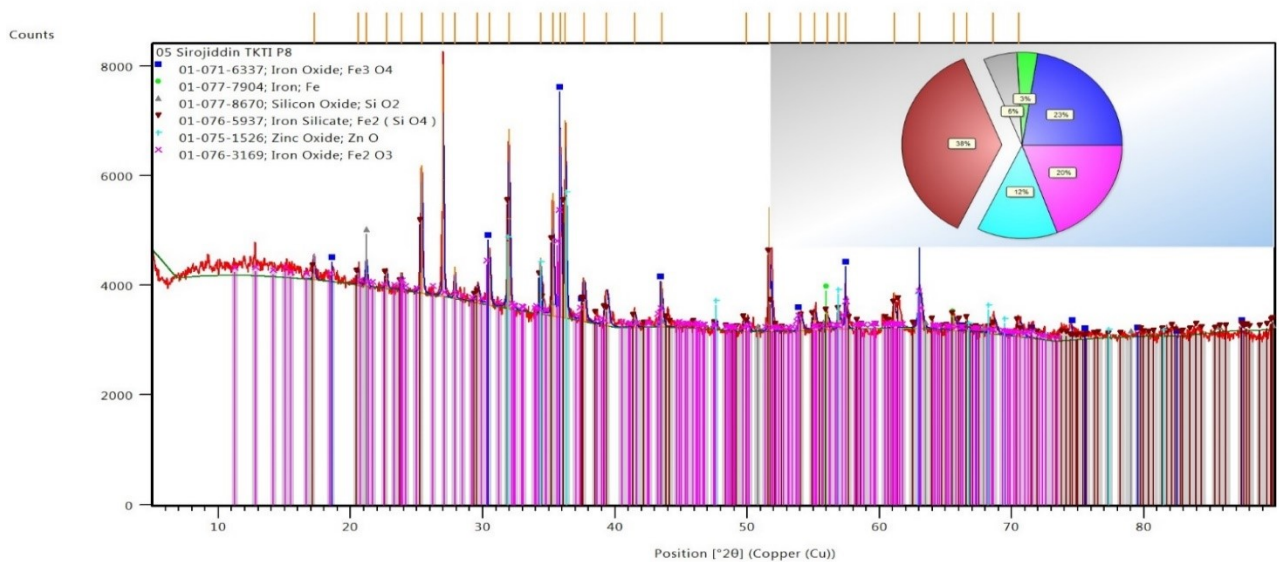
Pic.3. Diffractometric analysis results of iron-containing tail

From Fig. 3. it can be seen that in the iron-containing waste CSP-2, iron oxide compounds in the form of hematite make up 20%, silicon dioxide 5%, iron in the form of magnetite - 23%, ZnO - 12%, iron - 3%. $Fe_2(SiO_4)$ – 38% Based on the analysis results, it can be concluded that the tailings after enrichment of slags from copper production CSP-2 are iron-containing raw materials for the production of iron pigment Fe_2O_3.

Element	Weight .%	Sigma Weight.%
O	31.06	0.33
Mg	0.74	0.08
Al	3.94	0.11
Si	17.26	0.19
S	0.80	0.07
K	2.53	0.10
Ca	1.77	0.10
Ti	0.33	0.10
Fe	40.51	0.37
Zn	1.05	0.21
sum:	100.00	

Рис. 4 Results of spectral analysis of the first sample, presented as a spectrum (Energy dispersive spectroscopy (EDS))
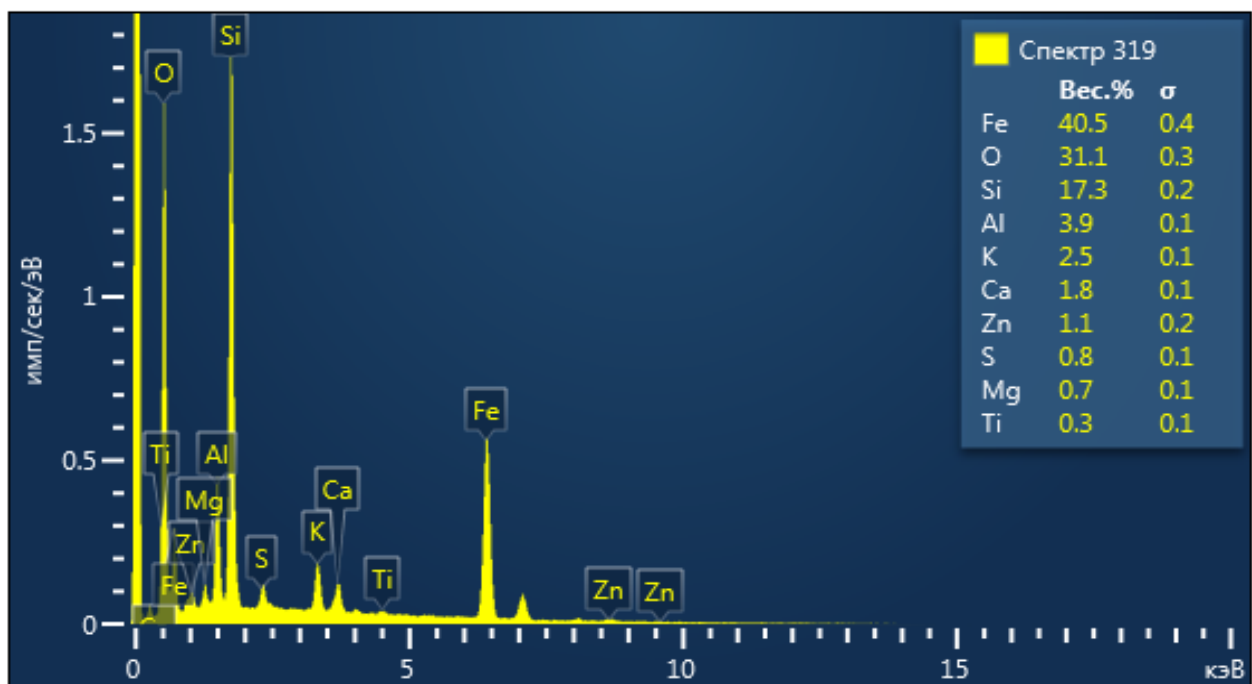
Conclusion

According to the results of chemical and mineralogical analyzes of the material under study, the following are obtained:

1. The accumulation of unprocessed waste causes potential economic and environmental problems: waste of resources and pollution of the environment: water, forests, land, atmosphere, etc.

2. From literary analysis it has been established that the total volume of waste (tailings, slag, sludge, dust, etc.) already accounts for more than half (about 60%) of the volume of the original iron ore, and 11-12% of subsequent processing, metallurgical, metalworking, etc.

3. It has been established that iron-containing waste (tailings) of CPF-2 contains iron and its compounds more than 50% or more.

4. It has been established that iron-containing waste MOF-2 contains iron oxide compounds in the form of hematite 49%, silicon dioxide 33-34%, CuO - 0.6%, ZnO - 1.56%, S - 0.8%.

5. It has been established that tailings after enrichment of copper production slag CPF-2 is an iron-containing raw material for the production of iron pigment Fe_2O_3.

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