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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

EXTRACTION OF INDUSTRIAL IMPORTANT METALS (Co, Cu, Zn, Mn) FROM PROCESSING DASHKASAN IRON ORE TAILINGS

Specialty: 3303.01 - Chemical technology and engineering

Field of science: Technical science

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The work was performed at the Institute of Catalysis and Inorganic Chemistry named after academician M. Nagiyev of the Azerbaijan National Academy of Sciences at the laboratory "Processing of non-ferrous metal mineral raw materials".

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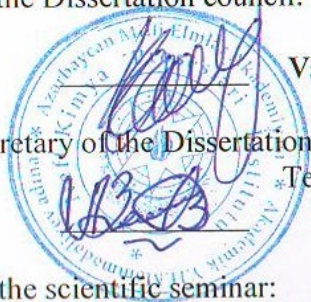
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INTRODUCTION

The relevance of the theme and the degree of development. Relatively low, sparse, difficult operating environment and poor environmental conditions in the country, which have many precious metal deposits in Azerbaijan, don't allow the use of classic operating technologies. On the other hand, relatively poorer areas remain not used, because the classically exploited fields are gradually decreasing due to the studied metal rich areas. Sometimes the concentration of metals in wastes is equivalent to the content of metals in the poorer areas of the ore.

More than 90 million tons of iron ore have been processed in the mines of the Dashkesan Mining Company OJSC (2001, the Azerbaijan Mining and Metallurgical Combine). During these years, as a result of processing iron ore, 46.5 million tons of waste have been extracted. The wastes from both dry and wet enrichment of iron ore of the factory were collected in an area of 42 ha landfill. These wastes are subjected to natural washing up by rain and snow and it is a cause the migration of metals to the environment by wastewater, which violates the environmental standards of the area. As a result of years of exploitation of the Dashkesan iron ore deposit, the waste collected around the bed can be used as a source of many precious metals. Calculations based on the approximate size, specific weight and other parameters of the sand dumps showed that its total mass is at least 20 million tons.

Currently, there are several types of mineral wastes in the Dashkesan iron ore field: quarrying rocks, waste ore deposits, iron ore enrichment plant, cobalt and alunite deposits. These wastes contain significant amounts of heavy and non-ferrous metals (Co, Cu, Mn, Zn, Cd, Pb, Fe, Al, Cr, V, etc.). These highly enriched wastes are not considered to be profitable to work with existing chemical processing methods. Therefore, the development of new processing methods that utilize natural resources efficiently, expanding the raw material base for the production of valuable components, is at the forefront. For this reason, poor areas, including waste treatment, may be considered more suitable for underground or

ball washing. Special attention was given to the widespread use of local raw materials, primarily ore and waste products within the Strategic Roadmap for the Development of Heavy Industry and Engineering in the Republic of Azerbaijan, adopted on December 6, 2016 (Article 7.2.3; Priority 2.3; Measures 2.3.2; 2.4.1; 1.1.4., etc).

The aim of the presented dissertation is to develop priority areas for the integrated processing of iron ore wastes. At present, the mining industry is facing the challenge of acquiring high-quality products with innovative and scientifically based ways. In this regard, the carried out scientific research works are actual on the condensation field, of removal of Co, Cu, Zn and Mn from iron ore-processing wastes and are relevant to the implementation of the Industrialization Program of the Republic of Azerbaijan in addressing the existing problems considered in the Road Map.

The mineralogical and chemical composition of the Dashkesan ore basin is completely different from the enrichment products in foreign countries. Up to this date, there is no industrial processing technology contain for cobalt, copper, zinc, manganese and aluminum wastes, and the issue of processing such raw materials remains actual. The presented work is dedicating to solving this problem.

The object and subject of the research is the wastes acquired from the processing of the Dashkesan ore basin and the enrichment of the iron ore in the Koshgar River valley.

The purpose and objectives of the study. Determine the legalities of economically viable metals washing in the composition of mineral wastes from the Dashkesan iron ore processing, the development of high-tech metals for the prevention of environmental contamination and the development of precious metals by working on the method of extracting valuable components. The following tasks have been proposed and implemented to achieve this goal:

- determination of economically viable metals as part of recyclable mineral waste and organization of their production processes;

- definition of the granulometric, chemical and mineralogical composition of wastes;

- to find the sources of sulfuric acid needed for decomposition of waste;
- to study the kinetic properties of lubricants with a weak solution of sulfuric acid and the mechanism of transfer of valuable components into the solution;
- definition of factors contributing the transition of metals to the solution from waste;
- to study changes in the surface properties of the minerals included in the waste;
- extraction of valuable components in the form of selective concentrates separately by changing the pH of the solution;
- to study the mechanism of sorption and condensation of metals with a low content of solid particles using natural sorbents (zeolite Aydag modified with monoethanolamine);
- development of technology for the extraction of several precious metals (Co, Cu, Zn, Mn) from the waste of iron ore enrichment and the creation of mobile technological equipment;
- calculation of economic profit is based on current selling prices for metals produced on the basis of the estimated metal reserves in waste.

Research methods. The chemical, geochemical, X-ray (RFA), thermal (DTA), and infrared (IR), energy dispersion spectral (EDS) methods were used during the work.

Basic provisions for defense. Determination of economically profitable metals removed from the enrichment wastes of the Dashkasan iron ore; study of efficient parameters and modes of removal of Co, Cu, Zn, Mn from wastes; obtaining selective concentrates using from arsenic and iron purification, hydrolytic sedimentation and sorption methods of precious metals (Co, Cu, Zn, Mn) that have passed into the solution; development of technology for the extraction of a number of precious metals from the wastes by means of a solution and the creation of a mobile technological complex.

The scientific novelty of the research. For the first time, the regularities of washing metals (Co, Cu, Zn, Mn) containing weak sulfuric acid were determined. The kinetic parameters of the

composition of the components into the solution and the selective deposition of metals in the solution were determined at room temperature.

The dissolution conditions of arsenic and iron in the form of technological solutions and non-ferrous metal ions in the form of separate concentrates were studied, and the content of the obtained precipitation was confirmed by methods of physicochemical analysis.

The method of selective removal of non-ferrous metal ions from the technogenic solutions through monoethanolamine modified zeolite has been developed, the mechanism of sorption has been clarified, and it is known that the sorption of Co^{2+} , Cu^{2+} , Zn^{2+} , and Mn^{2+} ions occurs through two mechanisms: ionic exchange and complex formation. In addition, the sequence of ion sorption was determined.

The effective parameters and modes of extraction of precious metals from the tailings of the treatment plant using raw materials from other useful deposits of the Dashkesan ore basin (Zeylik-Alunite deposit, cobalt bed in North Dashkesan, marble limestone) are determined.

Theoretical and practical significance of the research.

Based on the studies, the optimal method for the integrated processing of iron ore from the enrichment of iron ore is proposed. The method is based on studying the conditions for the decomposition of wastes with weak sulfuric acid, the transfer of valuable components into a solution, and the removal of solutions by precipitation and sorption. A laboratory mobile pilot device for the extraction and concentration of precious metals, such as Co, Cu, Zn, Mn, from multicomponent solutions is presented. As a result of research, for the first time in Azerbaijan, a technology was developed to extract useful metals from ore wastes without harming the environment.

As a result, a technological scheme of environmental protection was proposed that provides integrated waste management, which is one of the global challenges of the modern era.

The personal contribution of the author. The current state

of processing low-grade ore and tailings with non-ferrous metals, statement of the problem, purpose of work, ways to achieve this goal, justification of the method used, sampling from complex mining operations, organization and conduct of experiments, preparation of articles and abstracts, was performed by the author. and sometimes with his presence

Approval and application. Based on the results of the completed thesis in the field of theoretical and applied research, 15 scientific papers were published: 7 articles and 8 abstracts. Dissertation materials were discussed in the following scientific conferences:

Republican scientific conference dedicated to the 80th anniversary of the Institute of Catalysis and Inorganic Chemistry named after academician M. Nagiyev (Baku, November 2016); Turkey-International Chemical Congress (Ankara, September 2017); International scientific conference dedicated to the 85th anniversary of academician R.A.Aliyeva (Baku, November 2017); Azerbaijan Technical University, Baku Steel Company, 2nd International Scientific and Technical Conference on Problems of Metallurgy and Materials Science (Baku, November 2017); International Symposium "Environmental and engineering aspects for sustainable living" (Hannover, december 2017), XIII International Scientific Conference "Actual Problems of Chemistry" for doctoral students, masters and young researchers dedicated to the 96th anniversary of Heydar Aliyev (Baku, 2019); I International Scientific Conference "Actual Problems of Modern Chemistry" dedicated to the 90th anniversary of the Institute of Petrochemical Processes (Baku, October 2019), Azerbaijan and Turkey Universities: Education, Science, Technology (Baku, December, 2019).

Name of the organization where the dissertation is performed. The dissertation research was carried out in accordance with the scientific program of the Institute of Catalysis and Inorganic Chemistry named after academician M. Nagiyev of ANAS (State Registration No. 0115 Az 2099).

Structure and scope of work. The dissertation consists of an introduction, 4 chapters, results and a list of 176 references. The

work consists of 187091 (introduction 10999, Chapter I 69011, Chapter II 25117, Chapter III 44021, Chapter IV 33660, results 4283) marks and contains 28 tables, 22 charts and 30 drawings.

The relevance of the dissertation is justified; the purpose of the work, the provisions to be defended, the scientific novelty and practical significance of the work are explained.

The first chapter presents an analysis of the literature materials on the modern methods and technologies of poor ore and waste processing in accordance with the content of the dissertation and analyzes different areas of processing. Also, this chapter investigates the presence of ionic forms in the solution depending on the pH of the metals (Fe, Mn, Cu, Zn, Co, and Al), which are in the composition of man-made waste. It also provides information on the selective removal of metals from man-made solutions.

The second chapter is called methodological section. It describes the characteristics of iron ore waste, the research object, the granulometric, chemical and mineralogical composition of the material, the course of the experiments and the description of the facilities used.

Chapter three. Dashkesan is dedicated to washing iron ore waste and extracting metal concentrates from technological solutions. It also presents the results of research on metal and arsenic removal from metals.

The fourth chapter is devoted to the study of the sorption of metal ions (Co, Cu, Zn, Mn) from waste washing solutions by natural zeolite which was modified by monoethanolamine.

The dissertation concludes with a summary of the research work performed, with a list of key findings and references to the literature.

BASIC CONTENT OF WORK

Literature review on modern methods and technologies of oxidized poor ore and waste processing.

Based on the literature review, it was found that the most effective way for removing industrial metals (Co, Cu, Zn, Mn) from the Dashkesan iron ore was the method of heated and underground washing with weak sulfuric acid solution. It is also shown that the Dashkesan ore basin emissions are different from other types of wastes due to their unsatisfactory content (because of occurring Co). The physical and chemical parameters of processing and washing of this type of waste and the selective separation of metal ions in the solution have not been fully understood in world practice.

The study of the presence of metal ions in the solution, depending on the pH, showed that the elements produce different types of hydroxycomplexes depending on the amount and metal content in the solution.

Characteristics, research object and methodology of Dashkesan iron ore waste.

The Dashkasan iron ore deposit is located on the northern slope of the Lesser Caucasus. State-approved iron ore reserves are 270 million tonnes. The Dashkasan Mining and Processing Company (OJSC) currently carry out exploitation of this field. The main commodity products manufactured by the Company are iron ore concentrate and fine iron ore. The factory produces about 500 kg of finished products per 1 t of ore¹.

The factory produces about 4-5.000 tons of iron ore per day. In 2018, OJSC produced 1.7 million tonnes of iron concentrate¹. The concentrate is produced by two methods - dry and wet (wet) magnetic separation.

During the enrichment, the useful components (Co, Cu, Zn, Mn, Al) contained in the iron ore are removed along with the ore, and these elements remain in the concentrate, while the other is disposed of as a waste treatment plant.

¹ <http://www.kepeztv.az/news/id/4340>

Whereas the main macroelements of waste are aluminum, calcium, iron, and silicon, microelements of interest are precious metals: cobalt, copper, zinc, manganese, lead, iridium, zirconium and arsenic and gallium.

Table 1 lists the physical and mechanical parameters of wastewater enrichment. According to the granulometric composition, 70-90% of emissions are particles smaller than 0.3 mm.

Table 1.

Physical and mechanical parameters of waste and average granulometric composition, %

Areas	Humidity of samples. %	The weight of wet samples. g/cm ³	Dry specific weight of samples. g/ cm ³	medium granulometric composition (mm). %							
				+ 0.63	- 0.63	- 0.4 + 0.315	- 0.315 + 0.16	- 0.16 + 0.1	- 0.1 + 0.063	- 0.063 + 0.005	- 0.005
1	2	3	4	5	6	7	8	9	10	11	12
I Area	12.5	1.89	1.83	1.3	1.9	6.9	42.5	22.3	11.4	7.7	6.0
II Area	0.74	1.85	1.89	6.5	6.1	17.2	44.8	14.2	5.9	2.3	3.0
III Area	4.6	1.66	2.0	3.2	3.3	8.8	31.5	20.7	14.7	4.5	13.3

Low waste: Co - 27-60 g/t, Cu 432-517 g/t, MnO - 0.24-0.71 g/t, Zn - 155 g/t, Pb - 82 g/t, Y- 27 g/t, Zr - 28 g/t and As - 121 g/t. such components.

Minerological analysis of wastes contains hematite (Fe₂O₃) - 5.6%; quartz (SiO₂) - 13.3%; pyrite (FeS₂) - 1.5%; calcite (CaCO₃) - 12.8%; dolomite (Ca,Mg(CO₃)₂) - 3.0%; manganocalcite ((Ca,Mn)CO₃) - 2.0%; kaolinite (Al₂O₃·2SiO₂·2H₂O) - 12.2%; clinochlorine ((Mg,Fe²⁺)₅Al₂Si₃O₁₀(OH)₈) - 9.3%; andradite 3CaO·Fe₂O₃·3SiO₂ - 40.3%; that the mineral consists of minerals that are.

Table 2.

Chemical composition of medium age wastewater samples taken from different sites

Areas	Average content of substances, %										
	SiO ₂	Na ₂ O	MnO	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	SO ₃	CuO	TiO ₂	CoO
I area	47.5	0.54	0.71	15.5	8.06	22.76	3.27	0.88	0.01	0.037	0.51
II area	44.0	0.56	0.49	21.2	8.02	12.42	3.45	8.25	0.17	0.4	0.01
III area	58.0	0.60	0.36	11.7	5.1	9.1	3.85	9.2	0.05	0.55	0.01

Micronutrients detected by spectral analysis in the samples taken: Ti, Cr, V, Mn, Cu, Zn, Pb, Co, Ni, Ga, Ba, Mo, Zr, Y, Sc, Sr. The content of these elements in the waste varies between 0.0001-0.01%.

We observed the activity and weight loss of minerals in the residues and residues after their processing with acid using a thermal (derivative graph) analysis method. It was found that the weight loss in raw wastes is 2.1% of the total mass and 0.12% in the residue after washing.

Finally, the method of chemical solubilization and washing of materials in the ball form is shown. This chapter also provides information on laboratory percolation devices and enhanced wear-and-tear laboratory equipment.

Investigation of valuable components from solutions of Dashkesan mining ore dressing plant by percolation.

Initial experiments were conducted on the search for the type of solvent that ensures effective wear of metals in waste. Comparison of different solvents (NaOH, NH₄Cl, (NH₄)₂SO₄, H₂SO₄, CH₃COOH) showed that the most effective reagent sulfuric acid solution for wastewater treatment. Bear in mind that at least 600 l of H₂SO₄ (1 N) is required for the removal of 1 ton of waste (according to our calculations). The acquisition of sulfuric acid to prevent reactive reactions were carried out by electrolysis of K₂SO₄ and

Na₂SO₄ solutions, which is an alunite-processing product.

The known wastewater (0.3-0.5 kg) was loaded into the columns and washed with a drop of sulfuric acid (0.1-2 N) solution with different concentrations. Factors influencing the quality of the solutions obtained during the experiments were: dilution of the wash solution, time, pH, eH, and the number of valuable components in the solution. The pH, eH and free acids were monitored in productive solutions, and analyzed for cobalt, manganese, copper, zinc, iron and other metals. The determination of metals in the solutions was performed on a Bruker-S2 Picofox X-ray wavelength spectrometer. Washing of columns in two modes: (a) After removal of abrasive solutions, they are returned to the original acid level (pH = 1.5); b) Acid, which is known for its comparative study of the dynamics of metal excretion, is injected with fresh servings until complete washing of valuable components in waste. The experiments were carried out in a spiral-shaped laboratory unit with an underground burner and in pipes similar to a percolation column. In these experiments the weight of the waste was varied from 5 to 10 kg.

The sulfuric acid required for the washing was purchase on a custom-made diaphragm electro dialysis unit with a flow rate. The cathode was use as stainless steel plate and lead plate as anode. The azbest plate bound the area between the electrodes. When a 6 V voltage and 3 A current exceeds the voltage of 0.25 liters of potassium sulfate solution, an environment with a pH=12-13 (alkaline) solution in the cathode area produces a pH=2-2.5 (acid) solution.

The main issue of our research is the separation and consolidation of industrial essential metals (cobalt, copper, zinc and manganese) from these elements by chemical means.

Initial experiments were carried out at room temperature by monitoring the amount of copper dissolved in the droplet solution by 0.1-2 N sulfuric acid solutions (Figure 1).

As you can see, the concentration of copper in the solution increases when the acid content increases from 0.1 N to 2 N. Based on the comparison of numerous experiments and calculations, the acidity of the acid required for wear was taken by 1 N.

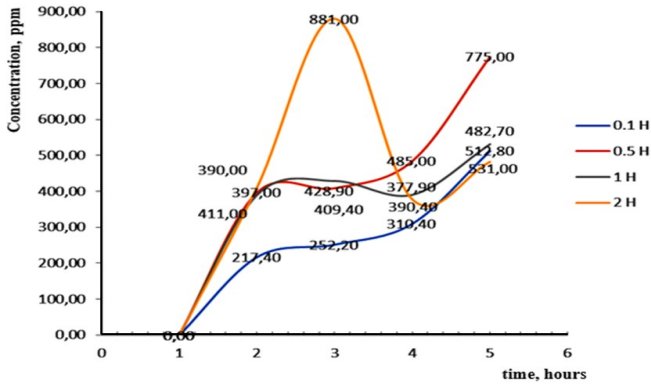


Figure 1. Copper concentration at different times depending on the sulfuric acid concentration

The results of the change in the metal content in the productive solution obtained during the drip modification depending on the amount of irrigation are given in Table 3. 30 % of the initial solution is used for soaking. The volume of the initial solution was reduced to 210 ml.

After washing six times, the pH of the solution gradually decreases from 8 to 6.0. The initial solution to increase the pH from 1.5 to 8 is the neutralization of calcium-containing minerals (calcite and dolomite) in the sulfate acid (1st stage irrigation). The amount of non-ferrous metals that pass into the solution is negligible. This may be due to the hydrolysis of the precipitated washed metal ions or their sorption on the solid-phase surface. In the 2nd and 3rd irrigations, the pH falls from 6.96 to 6.8. Such a drop in pH also makes a difference in the presence of non-ferrous metal ions. Cobalt 8.5; manganese 55; copper 5; zinc is 10 times more potent than previous irrigation.

In the fourth irrigation, the pH drops to 6.6 leads to a significant increase in the concentration of ions in the solution. At this acid level (pH = 6.6), the transition of iron to the solution is not yet observed. After the sixth irrigation, the equilibrium pH of the solution reached 6.

Table 3.

Change of metal content in the productive solution obtained during drip irrigation. (Watering time - 30 min, sample mass 500 g, initial pH = 1.5, H₂SO₄ acid volume, 300 ml of washing solution)

Number of experiences		Concentration of metals, mg/l						The pH of the solution after washing
		Ca	Co	Mn	Cu	Zn	Fe	
1	1 st stage irrigation	1537	0.89	2.39	0.93	1.5	3.41	8
2	2 nd stage irrigation	2700	1.52	40.59	2.97	4.09	—	6.96
3	3 rd stage irrigation	2891	7.57	134	4.66	14.98	—	6.8
4	4 th stage irrigation	3116	23.33	294	94.3	33.53	—	6.6
5	5 th stage irrigation	3272	31.23	392	228	43.39	—	6.4
6	6 th stage irrigation	4047	48.96	568	891	63.10	220	6.0

At the same time, the iron content in addition to cobalt, manganese, copper and zinc also increased (220 mg/l). Compared to the first irrigation, in the 6th irrigation metals are concentrated: cobalt 55 times, manganese - 238, copper - 958, zinc - 42, and iron - 64 times.

The next series of experiments were carried out with the change of the solvent time in days. The wash solution was stored in the percolator for 24 hours. The results are presented in Figure 2.

As can be seen, the concentration of precious metals increased in the solution, the pH decreased as the washing steps, and the contact time of the material with the acid increased. During the days 1-4 days, the pH decreased from 7 to 3.5, and the metal content in the solution increased rapidly. This can be attributed to the solution of fractions in thin layers in the upper layers of the waste particles.

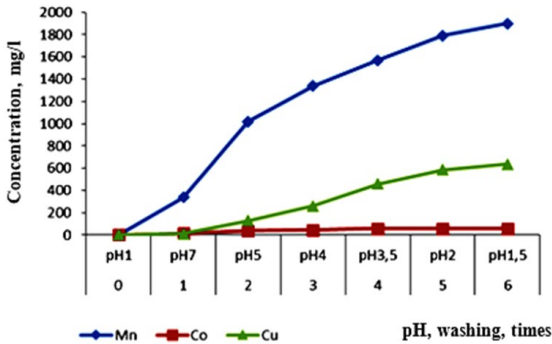


Figure 2. Time and pH dependence of the decomposition of the waste with 1 N sulfuric acid. (waste mass – 500 g, S:L = 5: 1, $t = 20^{\circ}\text{C}$, wear time is 24 hours)

Fractures 5 and 6 show little difference in the solubility intensity and the presence of metals in the solution. The low intensity of the reaction is due to the inability of the solvent molecules to diffuse into the waste particles due to the low conversion of the reaction product to the productive solution.

Changes in pH and oxidation redox potential (eH) during six washings (each time new acid is added) are shown in Figure 3 (a) and (b).

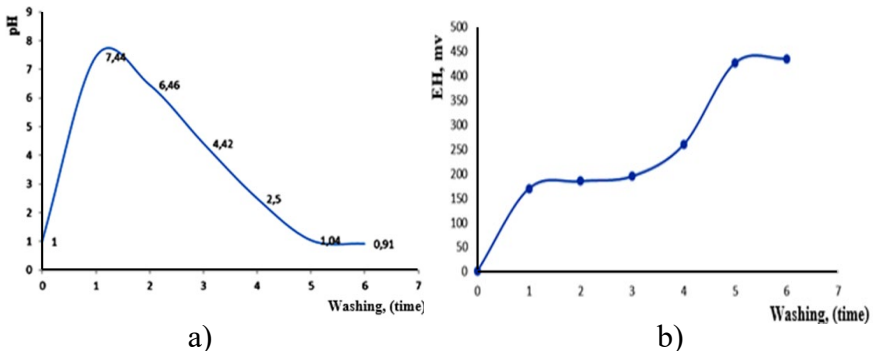


Figure 3. Change of solution pH (a) and oxidation redox potential (b) depending on the wash phase. (Mass of 500 g of waste, dilution of sulfuric acid used in washing - 1N, S: L = 5:1, temperature- 20°C , drip washing time - 24 hours)

As it can be seen, the pH = 0.95 of the detergent solution was increased to 7.44 during the first wash, and at subsequent washings pH = 0.91, and the oxidation-reduction potential of the process increased from 170 mV to 435 mV. This is explained by the fact that calcium-containing minerals (calcite and dolomite) in the first minutes neutralize the sulfuric acid solution (raising pH).

In subsequent washouts, there is an increase in the metal content with the increase of acidity as a result of hydrolysis of the sulfate compounds in the solutions obtained. In the fifth phase, the increase of the eH in the washing is due to the change in the metal and sulfur ions in the solution.

Subsequent experiments were carried out not with new detergents, but by re-injection of detergent into the percolation column each time. The results are shown in Figure 4.

In the first three periods, the pH of the solution increased from 3 to 6.5, while the metal dissolution in the wear increased gradually. The content of manganese and copper in the solution is 65.85 mg/l and 52.99 mg/l, whereas the cobalt and zinc content is 2.18 and 7.09 mg/l.

The passage of such small quantities of metals is due to the low acidity of the solutions. As can be seen from figure 4, the pH of the solution obtained at week 4 was 6.5. At this pH it is hydroxides, not iron, copper and zinc ion.

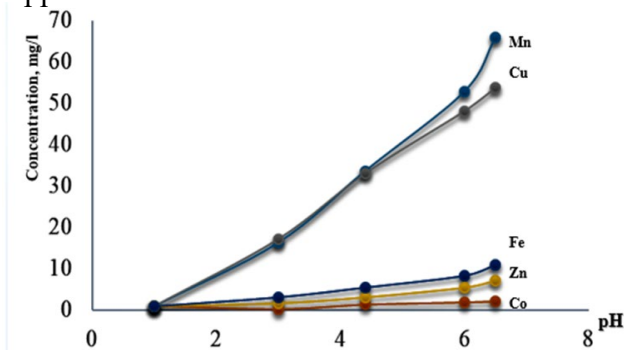


Figure 4. Depending on the pH of the metal solution with the release of the diluted solutions. (pH-3 of the solution obtained from the first cycle; 4.4 in the 2nd period; S: L = 1: 1)

The sediments obtained are caught by iron hydroxides in the lower part of the percolator. This can be explained by the gradual increase in the equilibrium pH of the solution from 3 to 6.5.

pH > 6.5 leads to a reduction in the concentration of certain metals: cobalt, copper, zinc and iron. In the lower part of the percolator (the solution) there is accumulation of ferrous iron hydroxides. It is in the form of iron - $\text{Fe}(\text{OH})_2$ and $\text{Fe}(\text{OH})_3$ precipitate in the solution at pH 5.2-6.5. At this pH, the Cu^{2+} , Zn^{2+} , Co^{2+} , Al^{3+} ions collapsed and adsorbed on the active surface of $\text{Fe}(\text{OH})_3$. Oxygen hydroxide amorphous phase of freshly collapsed three-phase iron has an absorption capacity of 150 mg-kv/g. Over time, as a result of dehydration of sediment (loss of OH group), the density of iron (III) hydroxide increases and its adsorption capacity decreases as the volume decreases. The presence of zinc and copper ions in fresh precipitated $\text{Fe}(\text{OH})_3$ in acidic medium (pH = 3) is not observed. The ions Zn^{2+} , Cu^{2+} , Fe^{2+} , Fe^{3+} , Co^{2+} , Mn^{2+} are dissolved in different ion and molecular forms depending on the cation density. Numerous experiments have concluded that it is not desirable to increase the pH of the solutions obtained from the washing of non-ferrous metal products to 3.5-4.

Oxidation of minerals contained in the waste and changes in surface properties. The change of mineral phases was investigated by investigating the diffractograms, microstructures, and IR spectra of samples in primary crude wastes, sulphate masses and cake from water solubility of sulfated mass.

Comparison of the initial emission and the diffractograms of its sulfated mass showed that there was no difference in the spectra. When new phases of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and epsomit ($\text{MgSO}_4 \cdot \text{H}_2\text{O}$) are formed, the intensity of arsenopyrite and calcite is lost, while andradite intensity is reduced. Quartz and Kuanite minerals have been show to be inert during the experiment and are practically insoluble. Comparison of 100-fold microphotos of waste and its sulfated mass shows that the structure changes in the sample under the influence of H^+ and SO_4^{2-} , leading to phase changes, cavities, and amorphous growth (Fig. 5). The results of EDS analysis showed that calcium was 19.56%, silica - 21.84%, iron - 17.11%,

aluminum - 10.30%, while magnesium, cobalt, nickel, potassium and sulfur were less than 3%, change between 0.9-2.78% (figure 5).

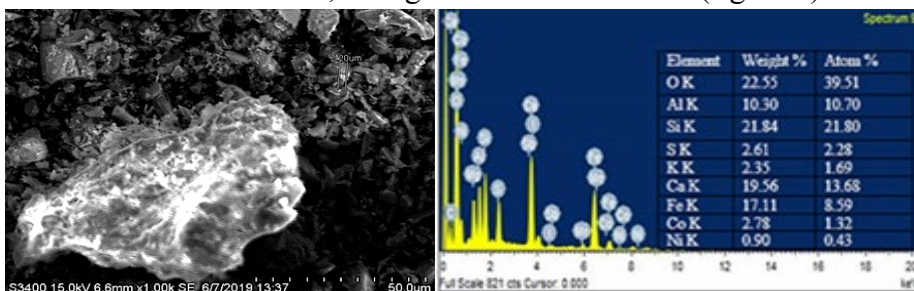


Figure 5. EDS analysis of sulfurized waste mass

The IR spectrum of the samples confirmed the presence of OH^- (3401 and 3542 cm^{-1} , $3000\text{-}3600\text{ cm}^{-1}$) and SO_4^{2-} groups (1116 and 602 cm^{-1}) in the sulfated mass.

Dashkasan iron ore waste washing and precipitation of metals from solutions.

Arsenic purification and selective concentrations of washing solutions by hydrolytic deposition were first performed in standard (Cu (II) , Zn (II) , Fe (II) , Fe (III) , Mn (II) , Co (II)) solutions. The obtained results were tested in technological solutions and the optimal process conditions were determined. The results of potentiometric titration of individual solutions of cobalt, copper, zinc, iron, aluminum and manganese sulphate with 0.1 N sodium hydroxide solution are shown in Figure 6.

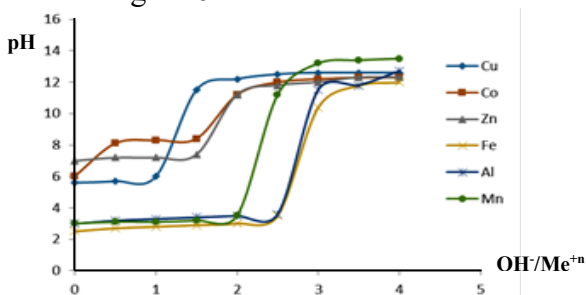


Figure 6. Dependence of pH on $\text{OH}^-/\text{Me}^{+n}$ ratio during potentiometric titration of metal ions with 0.1 N sodium hydroxide solution.

As you can see from the character of some, sediments of different compositions are forming in the titration of two- and three-valent metals sulfate salts with 0.1 N sodium hydroxide. In the 0.01M solutions containing Zn^{2+} , Cu^{2+} , Co^{2+} , Fe^{3+} ions, the initial starting deposition is at a pH of 6.4, and the complete deposition at 8.0; Initial deposition for copper pH = 6.3; full deposition at pH = 7.2, and starting sedimentation for cobalt at pH = 7.5; the complete deposition at pH = 9.2; Initial deposition for Fe (III) pH = 2.5; full deposition at pH = 4.57.

By IR spectroscopy and chemical analysis, it has been proven that the metal content of sulfate solutions ranges from 0.01 to 100 g/l with the basic salt of $MeSO_4 \cdot 3Me(OH)_2$, and of the highly saline solutions of $Me(OH)_n$ ($Me = Cu^{2+}, Zn^{2+}, Co^{2+}$).

Based on the chemical composition of the product solution and the presence of metal ions, we propose a simple and inexpensive method of refining iron and arsenic from lime, as well as non-ferrous metals in the form of selective hydrated sediments. Thus, since arsenic is highly toxic, its content in the solution should not exceed 0.05 mg/l. The most common method of separating arsenic from technological arsenic solutions is its deposition in the form of iron (III) arsenate (scorodite- $FeAsO_4 \cdot 2H_2O$) mineral.

The results of experiments on the deposition of iron and arsenic are presented in table 4, and the chemical composition of arsenic is shown in table 5.

Table 4

Results of deposition of iron and arsenic from solution

Consumption of $CaCO_3$. g/l	pH	Concentration of components in solution. g/l						
		H_2SO_4	Fe	Cu	Zn	Co	As	Al
0	1.0	31.2	13.63	0.64	0.089	0.007	0.23	1.9
30	2	24.5	3.54	0.64	0.089	0.007	0.05	1.9
45	2.8	8.2	1.45	0.64	0.089	0.007	0.02	1.9
60	3.2	4.1	0.1	0.63	0.086	0.007	0.01	1.8

As you can see, iron and arsenic, which collapses to pH = 3.2. The ions Zn^{2+} , Cu^{2+} , Co^{2+} , Mn^{2+} and Mg^{2+} remain in the solution.

Table 5

Chemical composition of sediment without neutralization at pH = 2.8 (%)

Components	CaO	Fe	S	Cu	Zn	Co	As
Mass share, %	31.2	35.5	30.1	0.09	0.004	0.003	0.086

The RFA of the faded green sediment obtained showed that it was amorphous. The sediment identification was carried out by IR spectroscopy and it was confirmed that the phase was $\text{FeAsO}_4 \cdot 2\text{H}_2\text{O}$.

At the next stage of processing, the research on separation of commodity copper-zinc concentrate in the filtrate without iron arsenic treatment was carried out. When the pH of the second step is removed by calcium carbonate at pH = 6 at 40°C, the two-valent copper, zinc, and slightly cobalt collapse.

Comparison of the masses of iron, copper, zinc and cobalt in the sediments showed that it is not necessary to raise the pH of the solution above 6-7 to obtain copper and zinc-rich sediments from technologically acidic solutions. Increasing the pH to 9 leads to an increase in the amount of cobalt and manganese in the sediment. Another half-chapter is devoted to the non-ferrous metal deposition from the solutions obtained from the sulphate treatment of cobalt ore.

Table 6.

Precipitate of valuable components in solution obtained from washing cobalt-rich ore with 1 N sulfuric acid (V=470 ml)

Quantity of elements in solution. mg/l									
pH	Sediment mass. g	Ca	Mn	Fe	Co	Cu	Zn	As	S
4.18		283	13.83	-	478	10.21	4.34	234.4	
Number of elements in the cake. %									
6	1.095	7.63	0.43	0.307	12.37	0.438	0.11	10.04	10.02
8.5	0.67	7.29	0.162	0.296	2.10		0.124	0.27	5.6
11	0.417		0.98	0.075	17.78			0.11	1.28
The percentage of deposition. %		99.5	99.52	99.90	99.66	99.5	99.0	100	98.8

Passing the mixture of cobalt high yield was achieved as a result of washing by 0.1 N acid mass obtained from sulfatization waste material containing the erythrits, cobalt and scutterudite minerals at 650⁰C. Composition of the components in the solution was as follows: Co - 2.17-3.43; Fe - 3.7-9.3; As - 1.305-2.77. The results of non-ferrous metals collapse in the solution obtained from the washing of cobalt-rich ore with 1 N sulfuric acid are given in Table 6.

The technological scheme for the removal of metals from the technological solutions in the form of selective concentrates is shown in Figure 7.

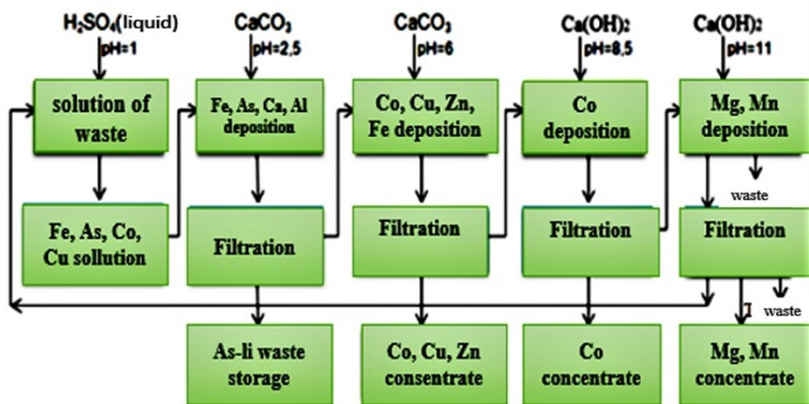


Figure 7. Dispersion of arsenic and metals in the form of selective concentrates from waste washing solutions.

Extraction Co, Cu, Zn, Mn ions from solutions by sorption.

The sorption of metal ions from sulphate solutions has been studied with the seagrass of the Aydagh field, which is widely used in industry. Zeolites are natural minerals that hold crystalline water, consisting of aluminium silicate of alkali and alkali-earth metals. The high sorption capacity, low cost, and wide dispersion in nature allow the ferrous metal ions in the zeolites to form and bind.

Natural zeolites are selective in low volume against non-ferrous metal ions. In order to increase their selectivity against non-

ferrous and rare metal ions, they perform the sorption characteristics of zeolites by modifying organic reagents. The selective sorbent of zeolite, which is modified by monoethanolamine (MEA), showing complex stability with non-ferrous metal ions, has been proven experimentally in the laboratory. In MEA that we use as modifiers, both the OH and the NH groups define the property of priority. The MEA is a special chelate-forming ligand that does not absorb alkaline earth metals.

Studies on the selective concentrations of copper, zinc, cobalt, manganese and aluminum hydrolytic sediments from the dumping solutions obtained from the Dashkesan ore basin showed that the ions in the solution did not completely collapse. It is interesting to note that the dissolved ions are absorbed by sorption.

The adsorption of non-ferrous metal cations (Co^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+}) was investigated in dynamic and static mode, both in natural and modified zeolite samples. Initially, the IR spectra of MEA and post-sorption samples were drawn, suggesting that the sorption is due to the formation of a metal complex with monoethanolamine on the surface of the zeolite. Complex reorganization is carried out through amino group nitrogen and alcohol group oxygen. The experimental results were calculated by the following formulas, expressed as SC (mg/g) and sorption rate (R, %) for zeolite.

$$SC = (C_0 - C_{eq}) \frac{V}{m}$$

$$R = \frac{C_0 - C_{eq}}{C_0} \cdot 100\%$$

Here SC sorption capacity (mg/g),

C_0 , C_{eq} - initial and equilibrium ions in the solution, mg/l

V-Volume of the solution, l

m- mass of sorbent, g

The Langmuir equation was used for the adsorption isotherm description.

$$SC = \frac{ST_{\max} \cdot k \cdot C}{1 + k \cdot C}$$

Here ST_{\max} is the final limit of adsorption capacity, mg/g
k- adsorption equilibrium constant,

The adsorption rate of Co^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} ions influences the pH of the solution. Thus, the electrolytic dissociation of the molecule changes depending on the pH. As the most valuable metal cobalt in technological solutions is its sorption from the solution. The pH dependence of the Ionite sorption capacity on the pH of the CoSO_4 solution is shown in figure 7.

According to the results obtained (figure 8), the optimal pH for sorption of cobalt solution may be between 6 and 8. In a strong acidic environment (pH = 1-2) sorption of cobalt is not observed.

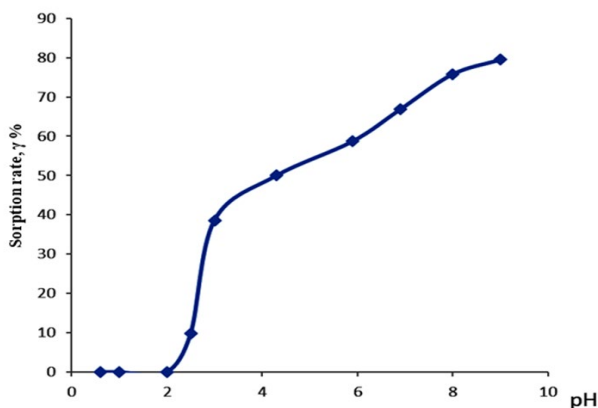


Figure 8. Dependence of ionic sorption rate on the pH of the CoSO_4 solution. ($C_{\text{Co}^{2+}} = 1$ g/l, contact duration is 1 day, mionite = 1 g, $V_{\text{sol}} = 0.1$ l.)

As can be seen, the low pH of the solution significantly affects the sorption characteristic of the ionite, to be more precise, the ability of the alkali groups in the zeolite to be altered by the influence of the H^+ ion is reduced.

In addition, initial experiments have shown that increased pH increases sorption rate of copper, zinc and manganese with modified zeolite in addition to cobalt. In a strong acidic environment (1 N H_2SO_4) the sorption of these metals does not occur. This is explained by the fact that in the acidic solutions these ions can be desorbed by

ionite.

Kinetic values of the Aydag natural zeolite beds and its modified monoethanolamine sample at the pH=6 ions of the Co^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} ions at different times are shown in Figure 9.

A comparison of the sorption capacities of the modified (a) and the unstated (b) zeolites showed that the adsorption of Co^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} ions for the first 15 min in the modified zeolite with MEA occurs at a high rate during the velocity towards the end of the sorption decreases and the equilibrium in the system is formed for all ions within 1 hour. At this time, 98.1% of Co^{2+} , 95.6% of Cu^{2+} , 94% of Zn^{2+} and 87% of Mn^{2+} are absorbed. However, during this time, 40% Co^{2+} , 33% Cu^{2+} , 19% Zn^{2+} and 17% Mn^{2+} were absorbed from the unchanged zeolite solution.

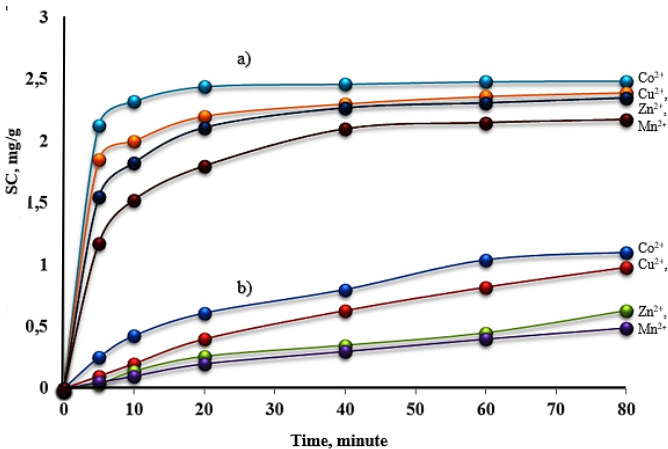


Figure 9. Depending on the contact duration of the sorption capacity of the ions of Co^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} . ($C_{\text{Me}^{2+}} = 100 \text{ mg/l}$, $\text{pH} = 6$, $t = 20^\circ \text{C}$, $m = 1 \text{g}$, $V_{\text{sol}} = 0.2 \text{l}$.)

A comparison of the sorption capacities of zeolites (Fig. 9) showed that the modification increases the activity of zeolite many times. When the initial metal content of individual solutions is 100 mg/l, the sorption capacity of zeolite modified by MEA is 2.5 mg/l for Co, 2.4 mg/l for Cu, 2.35 mg/l for Zn, and 2.2 mg/l for Mn. The results are four times higher than that of unmodified zeolite (Figure

9). The following range reduces the adsorption capacity of metal ions: $\text{Co}^{2+} > \text{Cu}^{2+} > \text{Zn}^{2+} > \text{Mn}^{2+}$. The sorption characteristics of each studied ion were calculated using the Langmuir and Freundlich equations. The results of the linear image change of the obtained experimental results in the Langmuir and Freundlich equations are shown in figure 10 (a) and (b).

The results obtained from the studies allow us to predict the sorption process through two mechanisms: ion exchange and donor-acceptor mechanisms.

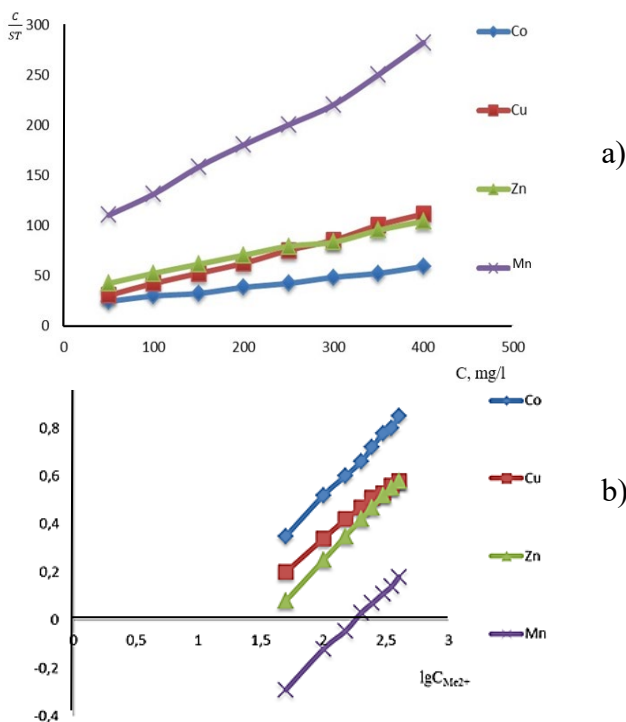


Figure 10. Langmuir (a) and Freundlich (b) isotherms for adsorption of Co^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} ions.

It is assumed that the ionization mechanism of Co^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} ions from the solutions is carried out by the substitution of the H^+ and OH^- groups in the seolide and by the heavy metal ions of Ca^{2+} , Mg^{2+} , K^+ , Na^+ cations in the zeolite. In addition

to the ionization effect, the formation of solid bonds between the modified zeolite and the metal cations (Co^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+}) can also cause sorption.

As a result of the research, a basic technological scheme for the removal of Co^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} ions from the Dashkasan iron ore enrichment was developed (Figure 11).

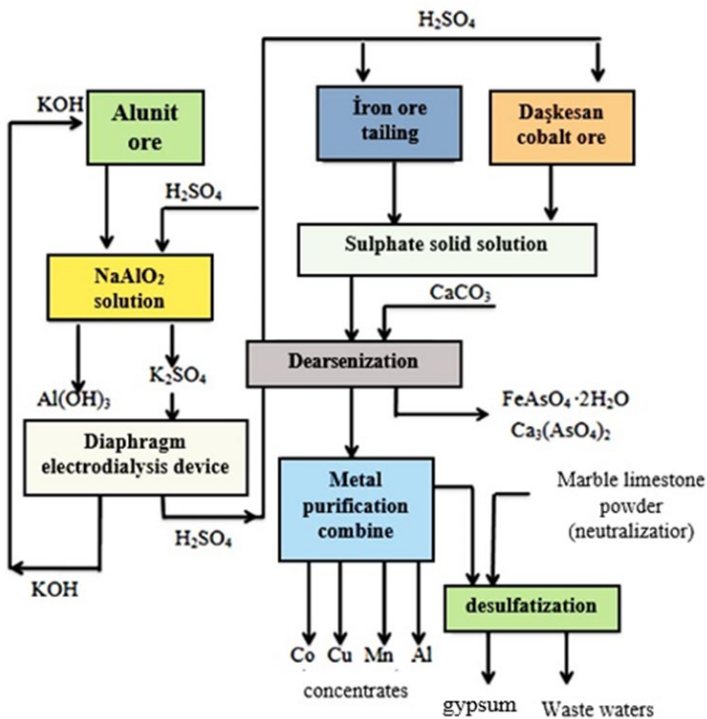


Figure 11. Principal technological scheme for extraction of cobalt, copper, manganese and aluminum from wastes of Dashkasan ore dressing combine.

The economic potential of the metals in Dashkasan ore dressing plant wastes (called "Beach" waste).

Due to the high potential of precious metal, Dashkasan mineral waste will contribute to the economic activities of Dashkasan Ore Purification OJSC as well as meet the needs of the relevant industry in the country, free from environmentally friendly and

heavy metals. to recultivate the area.

Even though 50% of the 20 million tonnes of waste can be extracted as a result of the proposed technology scheme, \$167.74 million could be economically earned by taking copper, zinc, cobalt and manganese concentrates separately. All these data indicate that the purification plant waste can be converted to cobalt, copper, manganese and manganese solution by recycling in complex form, and these elements can be obtained by hydrolytic deposition or sorption methods. After sanding, sand and gravel accumulated in the landfill can be used as building material.

CONCLUSIONS

1. Economically profitable metals in the wastes of the Dashkesan iron ore beneficiation were determined by preliminary chemical and mineralogical analysis of samples taken from different places and depths, as well as by studying the physical and mechanical properties of the waste [3]. Effective parameters and modes of extraction of valuable components from the tailings of the processing plant were determined by percolation solubility, diaphragm electrolysis, hydrolytic deposition and sorption [2].

2. The factors affecting the quality of the solutions obtained during the experiments were determined (solubility of the solution, washing time, pH and eH of the solution, S:L phase ratio, etc.). The electrolysis of the K_2SO_4 salt necessary for washing the waste was carried out from an alkaline solution of alunite at the Dashkesan-Zaglik deposit in an individually manufactured diaphragmatic electro dialysis unit with sulfuric acid,

3. The legitimacy of solubilization of the investigated waste with 1 N sulfuric acid was studied. During the six-step wash cycle with acidic acid, the pH at the initial stage increased from 0.95 to 7.44, and in the subsequent stages the acidity began to decline, and in the 6th phase, the pH dropped to 0.91. The oxidation-reduction potential of the process increased from 170 mV to 435 mV. The thickness of the detergent solutions in percolation columns (columns) was as follows: (sample I) pH = 2; Al-3.23 g / l; Fe-13.63 g/l; Mn-

1.89 g/l; Cu- 0.64 g/l; Co-5.8 mg/l; Zn-89.16 mg/l; (Sample II) pH = 4.8; Ca-223 mg/l; Mn-1383 mg/l; Co-478 mg/l; Cu-5.8 mg/l; Zn-4.34 mg/l. The percentage of metal release to the solution after washing 6 times was as follows: 65.9% Co; 30.5% Cu; 25.1% Zn; 17.5% Mn; 4.1% Fe; 1.82% Al [1, 6].

4. The methods of hydrolytic deposition and ionizing sorption were used to extract noble metals (Co, Cu, Zn, Mn) from technological solutions and to determine the degree of toxicity of arsenic. The method of systematic neutralization was used to obtain metals in the form of selective concentrates [1, 6]:

- when the pH of the solution was increased to 2.5–4 with the limestone, it was packaged and stored as a result of the collapse of arsenic in $\text{Ca}_3(\text{AsO}_4)_2$ and FeAsO_4 form (99.9%) [15].

- Cu, Zn, Co concentrate was obtained when the pH of the solution increased from 4.0 to 6.0.

- Coal concentrate was obtained when pH of the solution increased from 6.0 to 8.5, and concentrations of Mg and Mn at pH 11 were obtained.

5. Cobalt dissolution was achieved due to the washing of the mass obtained from the sulfuric acid at 650°C by the solidification of the waste material containing erythritol, cobalt and scutterudite minerals. The content of the components in the solution was as follows: Co - 2.17-3.43; Fe - 3.7-9.3; As - 1.305-2.77. The optimal conditions for the separation of the dissolved metals and non-ferrous metal ions together with the arsenic of iron were determined. It has been found that amorphous green pale green precipitate is dissolved at room temperature, and crystalline sediment ($\text{FeAsO}_4 \cdot 2\text{H}_2\text{O}$) at 90°C [11, 15].

6. Using the methods of physicochemical analysis, the oxidation of the minerals contained in the waste by sulfuric acid and the change in surface properties were studied, and it was concluded that sulfate acid increases the release of non-ferrous metals by increasing the mineral properties of the material in the waste [11].

7. The regularities of sorption of copper, zinc, cobalt and manganese ions with natural zeolites, modified by monoethanolamine, have been investigated following the extraction of non-

ferrous metal concentrates. It was found that zeolites can be used as an effective sorbent that separates Co^{2+} , Cu^{2+} , Zn^{2+} , Mn^{2+} ions. It was found that the adsorption properties of metal ions depend both on the pH of the solution and on the initial concentration of metal ions. The selective sequence of sorption of metal ions in amino-modified zeolite was as follows: $\text{Co}^{2+} > \text{Cu}^{2+} > \text{Zn}^{2+} > \text{Mn}^{2+}$. The adsorption results of the investigated systems have been investigated for Langmur and Freindlix isotherms. It has been found that the experimental results coincide with the Langmur area [9].

8. As a result of research, for the first time in Azerbaijan, the technology of extraction of useful metals from ore waste without harm to nature was developed. A test bench has been developed to simulate the underground sedimentation process under laboratory conditions. The implementation of the investigated technological scheme could bring in economic benefits of \$ 167.74 million, with the addition of cobalt, copper, zinc and manganese concentrates (metal concentrations reach 26-30%) [2].

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