

Article



Optimization of Conditions for Processing of Lead–Zinc Ores Enrichment Tailings of East Kazakhstan

Nazym Seksenova¹, Rudolf Bykov¹, Sergey Mamyachenkov², Gulzhan Daumova^{1,*} and Malika Kozhakanova¹

- ¹ School of Metallurgy and Mineral Processing, D. Serikbayev East Kazakhstan Technical University, Ust-Kamenogorsk 070001, Kazakhstan; seksen_nazym@mail.ru (N.S.); Rbykov@ektu.kz (R.B.); malika.kozhakanova21@gmail.com (M.K.)
- ² Department of Non-Ferrous Metals Metallurgy, Ural Federal University, 620002 Yekaterinburg, Russia; svmamyachenkov@yandex.ru
- * Correspondence: GDaumova@ektu.kz; Tel.: +7-777-396-12-47

Abstract: This article presents the results of studies of a low-waste technology for processing enrichment tailings using a combined enrichment–hydrometallurgical method. After washing the enrichment tailings from harmful products and reducing their size, multi-stage flotation of the crushed material of the enrichment tailings was carried out. The use of a new reagent in the flotation process was studied in order to ensure the maximum recovery of the main valuable components from the enrichment tailings. A new collector of Aero 7249 (Shenyang Florrea Chemicals Co., Ltd., Shenyang, China) type was used for the flotation. The recovery of valuable components was as follows: Cu, 6.78%; Zn, 91.69%; Pb, 80.81%; Au, 95.90%; Ag, 82.50%; Fe, 78.78%. Tailings of the flotation were re-enriched using a fatty acid collector (sodium oleate). Additional (reverse) flotation resulted in obtaining a product corresponding to the composition of building sand in terms of the content of valuable components of the waste rock. The studies of the conditions for processing the enrichment tailings of lead–zinc ore indicate the possibility of its optimization in order to maximize the involvement of waste in the production.

Keywords: enrichment tailings; flotation; metals; hydrometallurgy; lead-zinc ore; East Kazakhstan

1. Introduction

Processing of enrichment tailings includes solving technological and environmental problems. In this regard, in order to develop a low-waste technology for processing the enrichment tailings of non-ferrous metallurgy in Kazakhstan, it is proposed to consider the possibility of maximizing the use of enrichment tailings in the prevailing technological schemes employed in the metal industry.

Existing methods of processing the tailings usually include combined processes of enrichment, hydrometallurgy, pyrometallurgy and solvometallurgy. At the same time, the developed technological processes do not ensure the low-waste processing of enrichment tailings and their use in production [1].

Pyrometallurgical methods of processing the enrichment tailings are ineffective when working with low-grade raw materials—the costs of the products are higher than their market prices [2]. Another problem with traditional pyrometallurgical technologies is their low levels of environmental safety [3].

Palden et al. [4] studied the solvometallurgical method-selective leaching of lead and zinc from iron-rich jarosite in the zinc industry. Testing the various leaching agents showed that the presence of chloride anions is crucial for lead leaching. Ionic liquids, such as Aliquat 336 ((A336) (Cl)) and Cyphos IL 101 ((C101) (Cl)), were found to leach more lead and zinc after equilibration with HCl compared to other leaching agents. After leaching, the dissolved metals were recovered by selective distillation followed by precipitation with aqueous ammonia.



Citation: Seksenova, N.; Bykov, R.; Mamyachenkov, S.; Daumova, G.; Kozhakanova, M. Optimization of Conditions for Processing of Lead–Zinc Ores Enrichment Tailings of East Kazakhstan. *Metals* **2021**, *11*, 1802. https://doi.org/10.3390/ met11111802

Academic Editors: Petros E. Tsakiridis and Srecko Stopic

Received: 19 September 2021 Accepted: 5 November 2021 Published: 9 November 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Organic solvents are used in solvometallurgy to reduce energy, acid and water consumption, as well as to improve selectivity and reactivity. The disadvantage of this method is the need to use organic solvents, which complicates the technology of solutions purification after the leaching of enrichment tailings.

Leaching is the first step in the hydrometallurgical treatment of low-grade ores and enrichment tailings.

In [5], the treatment of Pb–Zn flotation tailings was carried out by leaching zinc with iron sulfate, which leads to a high yield of zinc with a recovery of 94.3%.

Various leaching agents (inorganic and organic acids, alkaline solutions and chelating agents) are used for the recovery of zinc from the tailings of the flotation of carbonate Pb–Zn ores. The maximum selectivity for Zn–Pb is achieved using sulfuric > hydrochloric > perchloric > nitric acids. It is also achieved using sulfosalicylic > citric > malic > formic acids. Sulfuric, citric, malic, sulfosalicylic and formic acids are recognized as the most promising leaching agents for the selective recovery of zinc from lead–zinc flotation tailings [6].

In [7], the authors investigated the possibility of using carbon materials (technical activated carbon and technical charcoal) as a catalyst in the leaching of zinc and copper from the tailings. Zinc and copper recovered from the mine tailings were determined after 2, 4 and 6 h. It is possible to recover more than 87% of Zn after 6 h of leaching with various sulfuric acid solutions. The addition of carbon-based materials increases the Zn recovery at high concentrations of sulfuric acid (1 M) from 89% to 99%. The use of technical charcoal significantly increases the extraction of Cu in the leach solution with a high concentration of sulfuric acid (1 M), from 41% to 61%.

Kursunoglu et al. [8] studied the choice of acid type for zinc recovery from enrichment waste using the Analytical Hierarchy Process (AHP) and ExpertChoice[®] 2000 (Expert Choice Inc., Arlington, TX, USA) software. The results showed that sulfuric acid is the most desirable acid, with a rating of 0.541, followed by citric acid (0.282) and oxalic acid (0.177). Citric acid can be used when the main environmental criterion rises from 7.8% to 75.3%.

Iron and sulfates were recovered from the tailings of lead–zinc–copper flotation using various inorganic acids in [9]. Recovery of iron and sulfate reaches 30% and 85%, respectively, at a HCl concentration of 4 M.

In the processing of enrichment tailings by the hydrometallurgical method, the adoption of low-waste technology and the maximum recovery of all non-ferrous and noble metals are issues in need of solution.

Bioleaching is of special interest among the methods for the recovery of metals from enrichment tailings. Gao at al. [10] described the influence of biological factors and the genetic data of microbes. After biological leaching of the enrichment tailings, residues may contain non-leachable heavy metals, therefore, post-treatment of the residues on the filter is also an issue.

The possibilities of increasing the rate of metal leaching by biological leaching in combination with other technologies are also being considered.

Recovery of metals from the lead–zinc mine tailings using bioleaching followed by the precipitation of sulfides was studied in [11]. Bacteria dissolved the metals from the tailings, and eventually 0.82% Pb, 97.38% Zn and 71.37% Fe were removed after 50 days. Thereafter, the metals were precipitated as a sulfide phase using sodium sulfide (Na₂S).

The Belousovskaya Enrichment Plant (Republic of Kazakhstan) achieved the following recoveries from old enrichment tailings using the combined enrichment and hydrometallurgical technology of leaching by *Thiobacillus ferrooxidans* microorganisms: Cu 75.66%, Pb 63.05%, Zn 75.53%, Fe 65.8%, Au 69.30% and Ag 67.36%. However, this technology, in spite of the high recovery rates, cannot be widely applied due to the complexity of the implementation and the cultivation of *Thiobacillus ferrooxidans* bacteria in the conditions of an operating enrichment plant [12].

The use of bacteria depends on the characteristics of the tailings being processed. The bioleaching process is ineffective due to the complexity of cultivating bacteria and using them in the required amounts.

Bagheri et al. [13] studied the recovery of sphalerite and pyrite from old tailings with a high zinc content. The effect of various flotation agents (collector, auxiliary collector, depressor, activators and foaming agent) on the characteristics of flotation was studied. The effect of pre-treatment of flotation pulp by cleaning or ultrasonic cleaning on the selective separation of sphalerite and pyrite minerals were also studied. Approximately 73% of the sphalerite is recovered from the accumulated tailings at a rougher stage. The foaming agents (MIBC and A65) has a synergistic effect and their mixture shows better enrichment results than using each foaming agent separately.

A solidification/stabilization method is used for the recovery of heavy metals from lead–zinc enrichment tailings [14]. Investigation of the properties of four typical chemical agents (Na₂S, NaH₂PO₄, TMT and Na₂EDTA) showed that heavy metals, lead and zinc in tails are stabilized more effectively when using TMT chelating agents. In this case, the stabilization efficiency of lead and zinc is 99.31% and 80.92%, respectively.

At the vast majority of enrichment plants processing sulphide lead–zinc ores, a rather difficult situation has developed due to the accumulation and storage of enrichment tailings. Due to the low content of lead and zinc and the low recovery of noble metals, the processing of enrichment tailings by traditional methods is not profitable and ineffective.

New methods beng needed for processing the solutions formed after leaching the raw materials under discussion and for the production of lead and zinc products, the original methods for the recovery of noble metals from leaching cakes are of interest for production.

When using the enrichment–hydrometallurgical method, it is necessary to carry out the enrichment processes, which requires the necessary equipment and flotation reagents. At the same time, the enrichment–hydrometallurgical technology makes it possible to ensure the maximum recovery of all valuable non-ferrous and noble metals into tailings, to solve the problem of low-waste technology of the entire mass of tailings with the inclusion of enrichment products in the production process.

To confirm the possibility of processing the enrichment tailings by the enrichment– hydrometallurgical method, a representative sample of the tailings of the Belousovskaya Enrichment Plant (Republic of Kazakhstan) was taken and examined.

Studies on the processing of enrichment tailings indicate the need for preliminary regrinding of stale tailings due to the presence of a significant number of intergrowths. Storage of old enrichment tailings in the aquatic environment of the storage facility results in significant changes in their mineral composition due to the accumulation of sludge products and products formed by the oxidation of reagents.

The surface of the main valuable components has different properties to those found in the natural state, which prevents their efficient processing by traditional enrichment methods. In this regard, it is advisable to carry out processes of preliminary preparation of the material of old tailings before their enrichment in order to increase the technological indicators of processing and reduce the consumption of reagents and materials.

2. Materials and Methods

A representative sample of lead–zinc ore enrichment tailings from Belousovskaya Enrichment Plant (East Kazakhstan region) was taken as the object of research. The ore sample was provided with a moisture content of 10%, and therefore it was dried and disintegrated in natural conditions. The sample of tailings was averaged by the ring-cone method.

After averaging and reducing the sample, a sieve analysis of the sample was carried out to determine the distribution of valuable components. Sieve analysis was carried out by hand-sieving the material using sieves with a mesh size of 10, 0.63, 0.5, 0.315, 0.071, and 0.044 mm.

Sieve analysis of the sample of old enrichment tailings is shown in Table 1.

4	of	10
---	----	----

Size Class, mm	+1	-1 + 0.63	-0.63 + 0.5	-0.5 + 0.315	-0.315 + 0.071	0.071 + 0.044	-0.044 + 0	Total
Yield, %	0.09	0.85	0.38	0.89	42.13	13.71	41.94	100

Table 1. Sieve analysis of the sample of old enrichment tailings.

Mineralogical, X-ray phase and chemical analyses were used to determine the material composition and content of the main valuable minerals and waste rock minerals in the enrichment tailings.

The characterization of noble metals in pyrite, chalcopyrite and sphalerite was established using mineralogical, X-ray phase and chemical analyzes.

Samples were analyzed using modern equipment: an automated Olympus BX53 (Olympus Corporation, Tokyo, Japan) microscope, an XRD-7000 X-ray diffractometer (Philips Corporation, Almelo. Netherlands) and an X-ray fluorescence analyzer (Malvern Panalytical Ltd., Malvern, United Kingdom). Grindability tests were carried out on tailings weighing 125 g in a 62–ML–A type ball mill. The ratio of solid, balls and water in the experiments was S:B:W = 1:1:9.

The washing of the clay-sludge part of the studied sample of enrichment tailings was carried out in measuring battery glasses with a supply of sulfuric acid with a pH of 3.

The following flotation reagents were used: H_2SO_4 , CaO, NaHCO³, liquid glass, Na₂S, CuSO₄, collector Aero 7249, sodium butyl xanthate, sodium oleate, butyl aeroflot, frothing agent T92.

Flotation was carried out on Mehnabor Technika (Research and Production Corporation "Mechanobr-Technika", St. Petersburg, Russia) and Denver 12 flotation machines (Jinshibao Mining Machinery, Shicheng, China) with chamber volumes of 0.75 L, 3 L and 5 L. The results were processed in Microsoft Excel (Excel 2016, Microsoft Corporation, Redmond, WA, USA) and MatLab (Matlab 9.8, Mathworks, Las Vegas, NV, USA) software.

3. Results and Discussion

The following processes were recommended when choosing the technology for the enrichment of the tailings from the Belousovskaya Enrichment Plant: regrinding degree, preliminary washing of the tailings, multi-stage collective flotation using a combined collector and non-standard technological parameters (Figure 1).



Figure 1. Recommended scheme for processing the old tailings of Pb–Zn ore enrichment.

As shown by previous studies of Belousovskaya Enrichment Plant tailings (East Kazakhstan Region) [15], their bulk is represented by a material with a complex chemical composition and a low content of main valuable components.

This feature of the material composition of the studied old tailings, as shown by subsequent research results, had a significant impact on the formation of the tailings processing technology. The chemical composition of the enrichment tailings is shown in Table 2.

Table 2. Chemical composition of the representative sample of the old enrichment tailings from the Belousovskaya Enrichment Plant.

Element	Pb	Zn	Cu	Fe	Au *	Ag *	Stotal	Ba	Al_2O_3	MgO	SiO_2	Albite	Muscovite	Kaolinite
Content, %(g/t)	0.07	0.2	0.07	6.94	0.32	3.18	5.01	0.62	13.9	4.34	28.9	3.6	27.64	8.71

Note: * The concentration of precious metals in g/t.

The phase composition of the main valuable elements of the sample is shown in Table 3.

Table 3. The	phase com	position of	f the main	elements o	of the enric	hment tailings sam	ple.
--------------	-----------	-------------	------------	------------	--------------	--------------------	------

	Pb			Zn		Cu				
Min and la	Conte	ent, %	El ann an ta	Conte	ent, %	El anno an ta	Conte	ent, %		
winerals	Absolute	Relative	- Elements -	Absolute	Relative	- Elements -	Absolute	Relative		
Galena	0	0	Sphalerite	1,23	100	Chalcopyrite	0.18	23.68		
Carakalita	0.29	100	Care i the earst ite	0	0	Azurite	0.26	34.21		
Carakonte	0.38	100	Smithsonite	0	0	malachite	0.3	39.47		
Oxidized	0.38	100	Oxidized	0	0	Oxidized	0.56	73.68		
Sulphide	0	0	Sulphide	1.23	100	Sulphide	0.18	23.68		
Total	0.38	100	Total	1.23	100	Total	0.76	100		

Gold and silver contained in the enrichment tailings are mainly associated with the following minerals: pyrite, chalcopyrite and galena.

The results of mineralogical analysis of the enrichment tailings sample are shown in Table 4.

Table 4. Mineral composition of the sample of old enrichment tailings.

The List of the	Minera Val	l Composition out the component of the c	Distribution of Mineral	
Main Components	Total	Inclu	uding	Grains in the Main
	Total	Aggregates	Free Grains	Components of families, /
Galena (Ga)	-	-	100	-
Sphalerite (Sl)	10	48	52	45% Ру; 33% Ру–Ср; 11% Ру
Chalcopyrite (Cp)	4	86	14	50% Sl–Py; Py
Pyrite (Py)	86	6	94	34% Sl; 25% Sl–Cp; Cp; 8% Sl
Total	100			

Due to the fine intergrowth of sulfide grains with grains of waste rock (Table 4), a series of experiments were carried out to select the optimal fineness of grinding the material of the tailings before their enrichment using the flotation process under conditions similar to those for ordinary ores of the Belousovskaya Enrichment Plant (Figure 2).

Test runs were carried out with the following grinding times: 10, 15, 20 and 30 min. The results of flotation at different fineness of grinding are shown in Figure 2.

The highest recovery of copper, zinc and silver was achieved when the fineness of the tailings was 73.4% of 0.044 mm, except for gold.





With a fineness of grinding of old enrichment tailings up to 73.4% in the bulk flotation concentrate, the content of the main valuable components was: Pb, 0.13%; Zn, 0.69%; Cu, 0.24%; and their recovery figures were: 27.17%, 57. 68% and 57.14%, respectively.

A similar grinding fineness of tailings before enrichment of the 73.4% class -0.044 mm was confirmed by previous studies [16].

To improve the indicators and conditions for the flotation of tailings, in our opinion it is advisable to clean the surface of mineral particles from oxidation products and flotation reagents to prevent them from deteriorating the technological indicators of enrichment and reducing the flotation rate.

The chemical composition of slurries differs significantly from the composition of the corresponding types of rock minerals in the initial ore samples (Table 3).

At additional grinding of collective concentrates and tailings, there is an increase in the content of fine classes of sulfide minerals of copper and zinc, pyrite, quartz and calcium oxide.

For this, pre-washing of the material in an acid environment was performed before grinding at a fineness of 73.4% of the class -0.044 mm, and flotation experiments under the same standard conditions were carried out using the Sabanin method [17].

The washing conditions were as follows: duration, 4 h; liquid to solid proportion, 7:1; environment, slightly acidic.

Losses of valuable components with discharge during washing were: Pb, 0.01 g/dm³; Zn, 0.024 g/dm³; Cu, 0.0013 g/dm³; and their recovery figures were: 0.03%, 0.03%, and 0.0043%, respectively.

Collective flotation was preceded by washing. Recovery of valuable metals to collective flotation concentrate with pre-washing was: Pb, 36.76%; Zn, 80.75%; Cu, 66.83%. This represents an increase compared to the experiments without washing (Figure 3). Comparative results of the recovery of valuable components from the tailings of the Belousovskaya Enrichment Plant by flotation with and without washing are demonstrated in Figure 3.

The use of pre-washing in a slightly acidic medium can increase the recovery of Pb by 9.59%, Zn by 23.07% and Cu by 9.69%.

Subsequent studies of the conditions for the flotation of old enrichment tailings were carried out on the washed material.

The collective flotation of old tailings of Pb–Zn ore was carried out in two stages (Figure 4).



Figure 3. Comparison of the flotation results with pre-washing in an acid environment and without.



Figure 4. Recommended scheme for the processing of old tailings at the Belousovskaya Enrichment Plant.

Conditions of the first stage of collective flotation were as follows: Na_2CO_3 , 1 kg/t; sodium butyl xanthate, 40 g/t; liquid glass, 400 g/t; long contact with Na_2CO_3 , 3 kg/t; fractional feeding of sodium butyl xanthat, 60 g/t; butyl aeroflot, 50 g/t; and foaming agent T9, 60 g/t. Flotation time, 10 min; pH 8. Time of the final flotation, 2 min (with feeding of sodium butyl xanthate, 20 g/t; and foaming agent T92, 20 g/t).

The tailings of the first collective flotation stage were subjected to long agitation with CaO (5 kg/t) and were then sent to the second stage of collective flotation.

Conditions of the second stage of collective flotation were as follows: Na_2S , 200 g/t; liquid glass, 500 g/t; fractional feeding of collectors sodium butyl xanthate and Aero 7249 (1:2), 500 g/t; and foaming agent T92, 50 g/t. Flotation time, 10 min; pH, 11–12. Time of the final flotation, 2 min (with feeding of sodium butyl xanthate, 50 g/t; and foaming agent T92, 20 g/t).

8 of 10

The combined use of the traditional sodium butyl xanthate collector and the new Aero 7249 collector were tested at the same time.

Two-stage collective flotation of tailings resulted in sufficiently high recoveries of the main valuable components. The results are shown in Table 5.

	NC 11 0/			C	Content, 9	%					R	ecovery	,%		
Product Name	Yield, %	Cu	Zn	Pb	Au *	Fe	S	SiO ₂	Cu	Zn	Pb	Au	Fe	S	SiO ₂
Collective flotation concentrate 1	22.82	0.28	0.66	0.2	0.7	18.02	17.56	6.44	64.96	74.83	46.02	53	59.6	77.08	3.35
Final collective flotation concentrate 1	2.76	0.08	0.16	0.16	1.12	15.38	9.99	7.32	2.25	2.22	4.33	10.27	6.15	5.3	0.46
Collective flotation concentrate 2	22.49	0.07	0.11	0.11	0.09	2.71	1.29	25	16.46	12.04	25.19	6.89	8.84	5.56	12.81
Final collective flotation concentrate 2	1.83	0.09	0.17	0.07	4.17	22.55	8.78	11.8	1.57	1.54	1.3	25.44	5.98	3.09	0.49
Total collective product	49.9	0.17	0.36	0.15	0.57	11.14	9.49	15.1	85.24	90.63	76.84	95.6	80.57	91.03	17.11
Collective flotation tailings	50.1	0.03	0.04	0.05	0.03	2.68	0.93	72.6	14.76	9.37	23.16	4.4	19.43	8.97	82.89
tailings (feed)	100	0.1	0.2	0.1	0.3	6.9	5.2	43.9	100	100	100	100	100	100	100

Table 5. The results of two-stage collective flotation of tailings.

Note: * Content of noble metals in g/t.

As can be seen from Table 5, the volume of old enrichment tailings remains significant, which may cause the need to return them to the existing tailings storage at the factory. Maintaining high volumes of stored enrichment tailings does not reduce their harmful impact on the environment. Therefore, it is necessary to reduce their volumes to a minimum level in order to exclude their second return to the existing tailings storage at the factory or the construction of a new store for the tailings of a two-stage collective flotation. Both of these options are not acceptable due to the additional costs for the construction of a new tailings store and the additional increase in the harmful environmental impact due to the new tailings dump.

Based on the above, studies were conducted to reduce the volume of tailings of twostage collective flotation by carrying out reverse collective flotation of waste rock minerals (muscovite, kaolinite, bassanite, gypsum, calcite and feldspars).

The conditions of reverse collective flotation were as follows: total flotation time, 12 min; consumption of $NaC_{18}H_{33}O_2$ (sodium oleate), 440 g/t; Na_2S , 500 g/t; liquid glass, 300 g/t; CuSO₄, 50 g/t; foaming agent T92, 150 g/t.

The results of reverse collective flotation are shown in Table 6.

	V:-14 0/	Content, %									Re	covery	,%		
	ffelu, /o	Cu	Zn	Pb	Au *	Fe	S	SiO ₂	Cu	Zn	Pb	Au	Fe	S	SiO ₂
Total reverse flotation concentrate	67.25	0.02	0.02	0.01	0.01	2.35	1.04	20	40.44	34.84	17.78	33.04	46.76	75.36	19.05
Reverse flotation tailings	32.75	0.03	0.04	0.05	0.03	2.68	0.34	85	59.56	65.16	82.22	66.96	53.24	24.64	80.95
Tailings of two-stage collective flotation	100	0.03	0.04	0.04	0.03	3.38	0.93	70.6	100	100	100	100	100	100	100

Table 6. The results of reverse flotation of collective tailings.

Note: * Content of noble metals in g/t.

Based on the results of the reverse collective flotation, the following can be noted: The total concentrate of reverse flotation will constitute a waste product of the enrichment due to the low content of the main valuable components;

The tailings of the reverse flotation are characterized by an increased content and recovery of silicon dioxide, which was 85% and 80–95%, respectively.

Therefore, it is possible to consider the possibility of using the tailings of reverse flotation as a commercial product that meets the requirements of the interstate standard GOST 8736-2014 for construction sand of non-metallic raw materials [18].

The consumption of reagents for processing the old tailings is shown in Table 7.

								Reagent	Consumption	1, g/t			
Operation Name	Operation Time, min	рН	H ₂ SO ₄	CaO	NaHCO ₃	Liquid Glass	Na_2S	CuSO ₄	Collector Aero 7249	Sodium Butyl Xanthate	Sodium Oleate	Butyl Aeroflot	Frothing Agent T92
Washing	240	-	50	-	-	-	-	-	-	-	-	-	-
Tailings Regrinding	13	-	-	-	1000	400	-	-	-	40	-	-	-
1 Collective Flotation	10	8–9	-	-	3000	-	-	-	-	60	-	50	50
1 Final flotation	2	-	-	-	-	-	-	-	-	-	-	20	20
2 Collective Flotation	8	12	-	5000	-	500	200	-	150	350	-	-	50
2 Final Flotation	2	-	-	-	-	-	-	-	-	-	-	20	20
Main Reverse Flotation	10	7–8	-	-	-	300	500	50	-	-	400	-	-
Final Reverse flotation	2	-	-	-	-	-	-	-	-	-	40	-	-
Total Reagent Consumption	34	-	50	5000	4000	1200	700	50	150	450	440	90	140

Table 7. Reagents consumption for tailings processing.

The technological balance of metals for old tailings processing at the Belousovskaya Enrichment Plant is shown in Table 8.

Viald %	Content, %							Recovery, %					
ffeld, /o	Cu	Zn	Pb	Au *	Ag *	Fe	Cu	Zn	Pb	Au	Ag	Fe	
48.70	0.19	0.38	0.16	0.56	5.39	10.14	86.78	91.69	80.81	95.90	82.5	78.78	
13.10	0.02	0.02	0.01	0.01	0.67	2.35	2.50	1.29	1.33	0.59	2.76	4.91	
38.20	0.03	0.04	0.05	0.03	1.22	2.68	10.72	7.02	17.87	3.51	14.74	16.31	
100	0.10	0.20	0.10	0.29	3.18	6.27	100	100	100	100	100	100	
	Yield, % 48.70 13.10 38.20 100	Yield, % Cu 48.70 0.19 13.10 0.02 38.20 0.03 100 0.10	Yield, % Cu Zn 48.70 0.19 0.38 13.10 0.02 0.02 38.20 0.03 0.04 100 0.10 0.20	Yield, % Cout Cont Cu Zn Pb 48.70 0.19 0.38 0.16 13.10 0.02 0.02 0.01 38.20 0.03 0.04 0.05 100 0.10 0.20 0.10	Yield, % Cu Zn Pb Au * 48.70 0.19 0.38 0.16 0.56 13.10 0.02 0.02 0.01 0.01 38.20 0.03 0.04 0.05 0.03 100 0.10 0.20 0.10 0.29	Yield, % Cu Zn Pb Au * Ag * 48.70 0.19 0.38 0.16 0.56 5.39 13.10 0.02 0.02 0.01 0.01 0.67 38.20 0.03 0.04 0.05 0.03 1.22 100 0.10 0.20 0.10 0.29 3.18	Yield, % Cu Zn Pb Au* Ag* Fe 48.70 0.19 0.38 0.16 0.56 5.39 10.14 13.10 0.02 0.02 0.01 0.01 0.67 2.35 38.20 0.03 0.04 0.05 0.03 1.22 2.68 100 0.10 0.20 0.10 0.29 3.18 6.27	Yield, % Cu Zn Pb Au* Ag* Fe Cu 48.70 0.19 0.38 0.16 0.56 5.39 10.14 86.78 13.10 0.02 0.02 0.01 0.01 0.67 2.35 2.50 38.20 0.03 0.04 0.05 0.03 1.22 2.68 10.72 100 0.10 0.20 0.10 0.29 3.18 6.27 100	Yield, % Cou Zn Pb Au* Ag* Fe Cu Zn 48.70 0.19 0.38 0.16 0.56 5.39 10.14 86.78 91.69 13.10 0.02 0.02 0.01 0.01 0.67 2.35 2.50 1.29 38.20 0.03 0.04 0.05 0.03 1.22 2.68 10.72 7.02 100 0.10 0.20 0.10 0.29 3.18 6.27 100 100	Yield, % Content, % Record Cu Zn Pb Au* Ag* Fe Cu Zn Pb 48.70 0.19 0.38 0.16 0.56 5.39 10.14 86.78 91.69 80.81 13.10 0.02 0.02 0.01 0.07 2.35 2.50 1.29 1.33 38.20 0.03 0.04 0.05 0.03 1.22 2.68 10.72 7.02 17.87 100 0.10 0.20 0.10 0.29 3.18 6.27 100 100	Yield, % Cou Zn Pb Au* Ag* Fe Cu Zn Pb Au* 48.70 0.19 0.38 0.16 0.56 5.39 10.14 86.78 91.69 80.81 95.90 13.10 0.02 0.02 0.01 0.07 2.35 2.50 1.29 1.33 0.59 38.20 0.03 0.04 0.05 0.29 3.18 6.27 100 100 100 100	NetworkNetworkYield, $\%$ ZuZnPbAu*Ag*FeCuZnPbAuAg48.700.190.380.160.565.3910.1486.7891.6980.8195.9082.513.100.020.020.010.072.352.501.291.330.592.7638.200.030.040.050.031.222.6810.727.0217.873.5114.741000.100.200.100.293.186.27100100100100100	

Table 8. Technological balance of metals for old tailings processing.

Note: * Content of noble metals in g/t.

4. Conclusions

Based on the research into the choice of technology for the processing of old tailings at the Belousovskaya Enrichment Plant, the following points were established:

- 1. The need to regrind the material of tailings to a fineness of 73.4% of the class -0.044 mm, which corresponds to the existing experience of processing similar products of technogenic products of enrichment.
- 2. Pre-washing of the source tailings in a slightly acidic medium increases the recovery of copper, lead and zinc by 42.3%, with minimal losses of these metals with washing drains.
- 3. The feasibility of a multi-stage collective flotation in order to maximize the recovery of valuable components in collective concentrates.
- 4. During the multi-stage collective flotation, it is necessary to consider the physical and chemical properties of the main valuable minerals (sulfide and oxidized forms) and the waste rock minerals, which are included in old tailings.
- 5. The need for simultaneous use of several collectors based on a combination of existing and new types of selective reagents.

- 6. Deep enrichment of the old tailings is feasible, with a maximum recovery of valuable components and a minimum yield (13.10%) of waste products.
- In order to maximize the recovery of valuable components from collective concentrates, it is advisable to process them hydrometallurgically with subsequent enrichment of leaching cakes.

Author Contributions: N.S. and R.B. designed the experiments; N.S., M.K. and G.D. performed the experiments; R.B. and S.M. analyzed the data; N.S. and G.D. wrote, reviewed and edited the paper. All authors have read and agreed to the published version of the manuscript.

Funding: Not applicable.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are contained within the article.

Conflicts of Interest: The authors declare no conflict of interest.

References

- Shadrunova, I.V.; Gorlova, O.E.; Zhilina, V.A. The new paradigm of an environmentally-driven resource-saving technologies for processing of mining. In *IOP Conference Series Materials Science and Engineering*; IOP Publishing: Bristol, UK, 2019; Volume 687, p. 066048. [CrossRef]
- Ceng, C.; Wang, H.; Hu, W.; Li, L.; Shi, C. Recovery of iron and copper from copper tailings by coal-based direct reduction and magnetic separation. J. Iron Steel Res. Int. 2017, 24, 991–997. [CrossRef]
- 3. Dimitrijević, M.; Kostov, A.; Tasić, V.; Milosević, N. Influence of pyrometallurgical copper production on the environment. *J. Hazard. Mater.* **2009**, *164*, 892–899. [CrossRef] [PubMed]
- 4. Palden, T.; Regadio, M.; Onghena, B.; Binnemans, K. Selective metal recovery from jarosite residue by leaching with acidequilibrated ionic liquids and precipitation-stripping. *ACS Sustain. Chem. Eng.* **2019**, *7*, 4239–4246. [CrossRef]
- Asadi, T.; Azizi, A.; Lee, J.; Jahani, M. Leaching of zinc from a lead-zinc flotation tailing sample using ferric sulphate and sulfuric acid media. J. Environ. Chem. Eng. 2017, 5, 4769–4775. [CrossRef]
- 6. Hussaini, S.; Kursunoglu, S.; Top, S.; Ichlas, Z.T.; Kaya, M. Testing of 17-different leaching agents for the recovery of zinc from a carbonate-type Pb-Zn ore flotation tailing. *Min. Eng.* **2021**, *168*, 106935. [CrossRef]
- Álvarez, M.L.; Méndez, A.; Rodríguez-Pacheco, R.; Paz-Ferreiro, J.; Gascó, G. Recovery of Zinc and Copper from Mine Tailings by Acid Leaching Solutions Combined with Carbon-Based Materials. *Appl. Sci.* 2021, 11, 5166. [CrossRef]
- 8. Kursunoglu, S.; Kursunoglu, N.; Hussaini, S.; Kaya, M. Selection of an appropriate acid type for the recovery of zinc from a flotation tailing by the analytic hierarchy process. *J. Clean. Prod.* **2020**, *283*, 124659. [CrossRef]
- 9. Sayilgan, E.; Karacan, G. Characterization and evalution of removal conditions of lead-zinc-copper flotation plant waste. *J. Eng. Sci. Des.* **2019**, *7*, 175–181.
- 10. Gao, X.; Jiang, L.; Mao, Y.; Yao, B.; Jiang, P. Progress, Challenges, and Perspectives of Bioleaching for Recovering Heavy Metals from Mine Tailings. *Adsorp. Sci. Technol.* **2021**, *6*, 1–13. [CrossRef]
- 11. Ye, M.; Li, G.; Yan, P.; Ren, J.; Zheng, L.; Han, D.; Zhong, Y. Removal of metals from lead-zinc mine tailings using bioleaching and followed by sulfide precipitation. *Chemosphere*. **2017**, *185*, 1189–1196. [CrossRef] [PubMed]
- 12. Sycheva, E.A.; Akylbekov, A.; Ushakov, N.N.; Kushakova, L.B. Combined Method for Processing Tailings of Polymetallic Ores. East Mining and Metallurgical Research Institute of Non-ferrous Metals. KZ Patent No. 5305, 15 October 1993. p. 3.
- 13. Bagheri, B.; Vazifeh Mehrabani, J.; Farrokhpay, S. Recovery of sphalerite from a high zinc grade tailing. *J. Hazard. Mater.* **2019**, *381*, 120946. [CrossRef] [PubMed]
- 14. Luo, Z.; Tang, C.; Hao, Y.; Wang, Z.; Yang, G.; Wang, Y.; Mu, Y. Solidification/Stabilization of heavy metals and its efficiency in lead-zinc tailings using different chemical agents. *Environ. Technol.* **2020**, *41*, 1–26. [CrossRef] [PubMed]
- Dryuchkova, O.A.; Bykov, R.A.; Mamyachenkov, S.V. The Ways of Development of Combined Hydrometallurgical Technology for Processing of Complex Ores' Beneficiation Tailings. *Mater. Sci. Forum.* 2019, 946, 564–568. [CrossRef]
- Mantsevich, M.I.; Malinsky, R.A.; Lapshina, G.A.; Khersonsky, M.I. Development of technologies for processing non-ferrous metal ores aimed at improving the environmental safety of mining and metallurgical industries. *Min. Inf. Anal. Bull.* 2013, 11, 74–81.
- 17. Mitrofanov, S.I. The study of minerals for enrichment. In *Textbook*; Gosgortehizdat: Moscow, Russia, 1962; p. 729.
- 18. GOST 8736-2014 Sand for Construction Works; Specifications: Moscow, Russia, 2015.