



SELECTING CIRCUIT PROCESSING SULFIDE RAW MATERIALS USING ALKALI METAL SALTS

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ABSTRACT

Polymetallic sulphide concentrates obtained by bulk flotation of ores. Removing the collective concentrate all valuable components (Cu, Zn, Pb, and others). 10-15% higher compared with the recovery in a selective concentrate, reduced scheme of enrichment and processing costs repartition. Collective concentrate of heavy nonferrous metals typically comprises: Cu₂S- chalcocite, CuS- covellite, CuFeS₂- chalcopyrite, sphalerite and wurtzite ZnS, pBS-galena, FeS₂ - pyrite, Fe₂O₃-Fe₃O₄-FeO - iron oxides, SiO₂- quartz, silica, Al₂O₃ - alumina, CaCO₃-MgCO₃- carbonates, as well as precious and rare metals. For the processing of sulphide concentrates and middling can use regenerable process reagent - soda ash Na₂CO₃. From a thermodynamic analysis of the reactions for HSC Chemistry Program¹ revealed that the most favorable scheme to form a metal oxide and with the binding of sulfur to sodium sulfate and release of carbon dioxide:



Thermodynamic analysis of the experimental determination of the beginning of the reaction temperature and firing the main sulfides of iron, copper, lead and zinc enable us to offer the following possible scheme for processing of collective concentrates.

"Firing sulphatization - leaching with water to extract the sodium sulphate - sulfuric acid leaching of the Ceck under oxidizing conditions - clearing iron from the solution and recovering copper and zinc from the solution - melting stage acid Ceck with extraction of lead and noble metals"

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The aim of the study is to examine the influence of technological factors on the firing charge of operations indicators drawn from the collective concentrate and soda, as well as the definition of the product yield according to the proposed scheme, and extracts of the main components.

We used bulk concentrate flotation sludge Malevsky ore deposits, applied methods of X-ray analysis of raw materials and products, gas chromatographic analysis on the content of carbon dioxide.

Key words: Bulk concentrate of nonferrous metal sulfides, Pyrite, Chalcocite, Galena, Sphalerite, Sodium sulfate, Sodium carbonate, Roasting, Sulfation, Oxidation leaching.

INTRODUCTION

Technology of complex processing of polymetallic raw materials of non-ferrous metals in recent decades, developed on the basis of electro thermal and autogenously melting processes (process Vanyukov A.V., flash smelting, etc.), Process intensive bubbling gases melts (the QSL process, Ausmelt, Isasmelt). The hydrometallurgical process is used mechanical activation and ultrafine grinding feedstock (Albion process), and to increase the reaction rate and the performance units - technology autoclave leaching and solution purification from impurities. Actual problems of the development of new technologies and roasting melting with alkali metal salts based on the thermodynamics of complex systems and experimental studies to determine the optimal conditions for conducting trials.

The article² noted that the technology for the processing of polymetallic ores analysis suggests the need to review the concept and purpose of the redistribution of wealth. Preparation of concentrates of ores monometallic associated not only with a significant loss of valuable components dissimilar in pyrite concentrates and tails, but the high cost of the selective enrichment and the use of toxic reagents in flotation. Already, the majority of ore mining and processing enterprises produced low-grade concentrates, which create technological and ecological problems in the plants or stored in dumps. The author believes that the promising direction of development is to limit the role of the repartition of the transfer of enrichment and separation problems metals metallurgical repartition. Tasks repartition enrichment can limit the separation of useful components from rock minerals or iron sulphide concentrates to produce a collective or industrial products. An example of a new technology for the low-grade lead-containing concentrates, dusts and copper-zinc middling's enrichment. The technological scheme includes redistributions oxidative roasting furnaces and tapping electro thermal melting with a small volume of exhaust gases and dust collection and disposal of sulfur dioxide system. The process employs a method of melting reaction, which should be in the mixture ratio provided by calcination of lead sulfide to sulfate at least 1: 2. Electro fusion allows to obtain crude lead, copper-lead matte and slag, wherein the zinc concentrates.

In³ it is noted that, in connection with the development of deposits and an increase in the volume of refractory ores difficult selective allocation of concentrates due to a significant proportion of aggregates and emulsion isomorphic inclusions of sulfides of iron and non-ferrous metals in waste rock. When ore dressing deteriorated quality zinc concentrates: zinc content decreased; the iron content increased to 10-14%, 2-4% copper, 6-10% silica, 4-8% lead, arsenic. With indicators of metallurgical production technology concentrates decreased deterioration of quality: reduced yield of soluble forms of zinc in the formation of poorly soluble iron and zinc silicate; deteriorated pulp division; complicated cleaning solution with the formation of an increased amount of Cecks.

We have previously⁴ was conducted overview of contemporary research and patents proposing concentrates melting technology and industrial products containing lead sulfide, copper, zinc, nickel, iron using KOH alkaline and NaOH, and alkali metal carbonates. Analysis of publications and experimental studies have shown that the most appropriate technology to the alkali metal salts proposed in D.A.Norrgran patent⁵. To remove the base metals, iron ore is enriched by flotation, and the concentrate is mixed with carbonate or bicarbonate is calcites. Consumption of alkali metal carbonate 10-20% by weight of the calcine, the firing temperature 550-650°C, the firing duration 2-4 hours. The calcine is mixed with water to separate the soluble sulphates of metals from iron compounds and other solid residue. Sulphate solution is filtered from particulate matter and non-ferrous metals recovered by precipitation or electrolysis.

Purpose of the study. The influence of the main technological factors during firing of the charge on the basis of the bulk concentrate and sodium carbonate to determine the distribution of non-ferrous metals and iron product flow sheet consisting of firing the charge - water and acid leaching Cecks - soda melting lead-free Ceck.

EXPERIMENTAL

Raw materials: For the experiments, select an existing collective concentrate flotation sludge Malevsky ore deposits and soda ash. X-ray diffract meter analysis of the concentrate was previously carried out on a diffract meter with Cu radiation, p - filter. Shooting conditions: U = 40 kV; I = 20 mA; range: 2000 counts; 2 with time constant; Shooting theta-2 theta; Detector 2 deg / min.

X-ray analysis showed the presence of phases in the concentrate of sphalerite ZnS, galena PbS, CuFeS₂ chalcopyrite and pyrite FeS₂. Gangue components (SiO₂, Al₂O₃, CaCO₃, MgCO₃) is not more than 0.5-0.7%.

Table 1: Mass fraction of elements and compounds, %

Element	Zn	Pb	Cu	Fe	Cd	As	Sb	Сообщ	Ba	Ni	SiO ₂	Al ₂ O ₃	Ca	Mg
% (weight)	27,29	10,58	5,99	17,30	0,13	0,10	0,13	35,96	0,31	0,004	0,24	< 0,1	0,005	0,1

Composition of concentrate as defined in the SPARC-1 spectrophotometer to compare the results of analysis of the product obtained by roasting, leaching and smelting to the original raw material composition. Analysis of Fe, Cu, Zn was conducted at an anode voltage of 25 kV, exposure time 20 sec. The results of analyzes on zinc and copper, practically coincide with previous analyzes on lead content below and slightly higher on the iron before the received data. Mass fraction of elements defined in the SPARC-1, %: Zn - 27,6; Pb - 5,9; Cu - 7.57; Fe - 21,6.

For the firing concentrate was mixed with a stoichiometric amount of soda ash. Soda ash is a chemical composition (wt.%): Na₂CO₃ - 92.8; K₂CO₃ - 3.8; Na₂SO₄ - 2.8; Cl - 0,5. Soda consumption is calculated from the sulfur content on the above listed ratios. Consumption of soda ash in the firing process of collective sulfide concentrate is determined sulfur content. According to the stoichiometry of the reaction, assuming that the sulfur in the sulphide polymetallic concentrates fully connected to higher and lower sulfides, soda consumption per 100 kg of concentrate can be calculated by weighing the sulfur content: $Q_{Na_2CO_3} = 3,3125 * S$ kg.

The charge share high sulfur concentrate amounted to 0,455 due to the high sulfur content (35.96%). The stoichiometric consumption of soda was chosen for complete utilization of sulfur in the concentrate sodium sulfate and subsequent recovery of soda. Finished blend was granulated after stirring at the laboratory granulator bowl with a diameter of 0.45 m bowl. When $n =$ speed of 60 rev/min and the slope $a = 45^\circ$. The finished pellets were dried in air and in oven at 110°C. Granules batch divided into classes with an average diameter of 2.5; 4.5; 8.5; 12 mm. Расход кислорода на реакцию с образованием оксида металла из пирита FeS₂ и халькопирита CuFeS₂ также можно рассчитать по сере: $O_2 = 1,75 * S$ кг.

Oxygen consumption reaction to form the metal oxide of the lowest type sulphides FeS, ZnS, PbS, is: $O_2 = 2,0 * S$ kg. In practice, you can take the theoretical maximum oxygen consumption for oxidation: $Q_{O_2} = 2,0 * S$, kg. When using the air flow will be in terms of: $V_{voz} = 6,74 * S$, m³.

When firing in the filter bed reactor, the CO₂ content of the gas was determined by a chromatograph "Gas Chrome - 3101". Temperature control in the furnace with Nick Chrome heater carried thermocouple PP-1 and potentiometer SH4501. The temperature in the additional reaction zone was measured with a thermocouple and digital DT-838 instrument. A portion of the materials weighed on electronic scales VLT-510-P (capto).

RESULTS AND DISCUSSION

Calcination granulated batch. Firing pellets was carried out with the sample of pellets of 50 * 10-3 kg at a rate of 55.2 liters of air with the use of probabilistic and deterministic planning experiments and the factors taken into account: temperature, air flow rate, the diameter of the granules, the firing duration. Controlled temperature, amount of supplied air blast, and the amount of carbon dioxide content in the flue gases. The exhaust gases are passed through a flask with distilled water to control the content of sulfur-containing gases. Only in some experiments in the exhaust gases at the beginning of the selection process was observed SO₂ in minor amounts, leading to a change in the control flask pH volume of 1.0 liters with pH = 7 to pH = 4.

Table 2: The experimental conditions and the results of the batch roasting

№ Experience	Temp. (°C)	Air consumption (l/h)	Pellet diameter (mm)	The firing time (min)	Desulphurization (%)
1	700,00	38,00	8,50	90	61,76
2	700,00	30,00	2,50	30	36,6
3	700,00	42,00	12,00	120	97,6
4	700,00	34,00	4,50	60	61,24
5	600,00	38,00	2,50	120	86,5
6	600,00	30,00	12,00	60	61,76
7	600,00	42,00	4,50	90	87,83
8	600,00	34,00	8,50	30	38,28
9	750,00	38,00	2,50	120	69,79
10	750,00	30,00	12,00	60	24,61
11	750,00	42,00	4,50	90	57,84
12	750,00	34,00	8,50	30	43,42
13	650,00	38,00	2,50	120	78,17

Cont...

№ Experience	Temp. (°C)	Air consumption (l/h)	Pellet diameter (mm)	The firing time (min)	Desulfurization (%)
14	650,00	30,00	12,00	60	31,26
15	650,00	42,00	4,50	90	82,96
16	650,00	34,00	8,50	30	26,8

To experience the best indicator №3 with desulfurization in Fig. 1 is a graph of changes in the carbon dioxide content in the flue gases from the reactor.

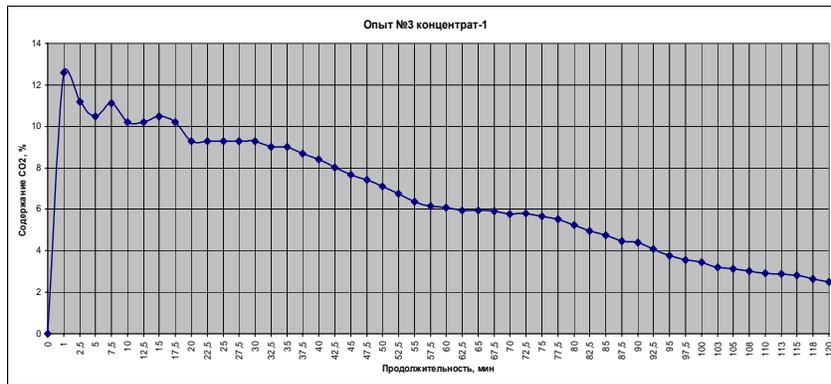


Fig. 1: Change of CO₂ in the flue gases of firing at 700°C

View reflects the change curve and concentrate desulphurization respectively sulphatization sodium carbonate is shown in Fig. 2. Practical results coincide with the theoretical curve represented by a polynomial of the second degree.

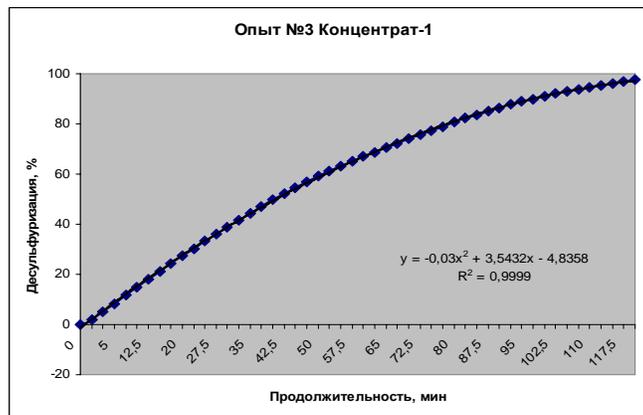


Fig. 2: The degree of desulfurization of the charge at 700°C

On the basis of the results carried out mathematical processing and obtained private (double) depending factors influence the firing process, the degree of desulfurization of the charge.

Table 3: Private depending roasting granulated batch

#	Influence factor	Type of private dependence	The dispersion coefficient of one A	The dispersion coefficient of the one in	The level of significance, TR1
1	Temperature (°C)	$Y_1 = 126,02 - 0,099 \cdot X_1$	6,019	0,006	1,002
2	Air consumption (l/h)	$Y_2 = - 85,407 + 4,016 \cdot X_2$	6,525	0,128	9,036
3	Pellet diameter (mm)	$Y_3 = 74,986 - 2,303 \cdot X_3$	8,655	0,833	1,005
4	The firing time (min)	$Y_4 = 17,127 + 0,56 \cdot X_4$	4,44	0,040	19,513

Given the level of significance of influence of factors Protodjakonova equation is as follows: $Y_{Pr} = 0,017 \cdot Y_2 \cdot Y_4$; coefficient of multiple correlation $R = 0,525$. For the purpose of imposing restrictions on the degree of conversion of 0 to 100% Protodjakonova equation entered into the exponent takes the form: $RE = 0,762$; $TRE = 4,403$. From the above sample levels of the factors were the most important air flow and duration of the experience. Less significant factors - the firing temperature and the diameter of the granules. Sample results for lower desulfurization experiment duration of 30, 40, 50, 60 minutes gives the following values of the partial dependencies.

The significance level of private depending $TR_1 > 2$ points to the need to consider the impact of this factor. Consequently, when the duration of the firing granulated batch at 60 minutes along with a factor affecting air flow rate and the duration of the experience and becomes the diameter of the granules. Estimated yield was $51.55 \text{ cinder} \cdot 10^{-3} \text{ kg}$. Practical way after weighing equals $47.53 \cdot 10^{-3} \text{ kg}$. The calculation does not take into account the dust, which has been taken from the cinder and settled on the walls of the reactor and the hot gas tubes. According to the balance calculation firing extracting all metals and alkali metal compounds is 99.99%, the degree of utilization of oxygen blowing is 82.4%. The recovery of carbon dioxide in the gases is equal to 97.9%, and agrees well with the practical result of the achieved degree of desulfurization of 97.6%.

Table 4: Private depending calcining the pellets at the sample 30, 40, 50, 60 minutes

#	Influence factor	Type of private dependence	The dispersion coefficient of one A	The dispersion coefficient of the one in	The level of significance, TR1
1	Temperature, C	$Y_1 = 107,287 - 0,081 * X_1$	3,893	0,004	1,715
2	Air consumption, l/h	$Y_2 = - 79,220 + 3,665 * X_2$	7,260	0,142	9,036
3	Pellet diameter, mm	$Y_3 = 75,942 - 3,379 * X_3$	4,310	0,415	8,827
4	The firing time,	$Y_4 = 17,127 + 0,56 * X_4$	7,659	0,119	5,103

Water and acid leaching of calcine. Calcine to the second stage of processing was subjected to leaching with water at a temperature of 30-35°C to recover soda and sodium sulphate residue. Leaching was conducted in a flask with a magnetic stirrer.

The weight of the original cinder experience № 3 was $47.53 * 10^{-3}$ kg, the volume of water 1 portion of $0.2 * 10^{-3}$ m³, 2-piece of $0.2 * 10^{-3}$ m³. Duration 30 minutes of leaching. The Ceck was filtered on a suction leaching - a filter and washed with water. Acidity in the first portion of the beginning of the leaching water was pH = 9-10, in the second stage a pH = 7-8. When washing the filter Ceck pH = 7, which is a sign of the complete washing of alkali metal salts. Keck-1 water leaching were dried at 120°C, 1-Ceckweight was $17.85 \text{ kg} * 10^{-3}$.

Keck-1 was leached with sulfuric acid at a concentration of 180 kg/m³ (density of the solution in 1110 kg/m³) at 60°C and air for aeration and oxidation of monovalent copper metal. The duration of leaching was 60 min, air flow rate at 3-fold excess of $3 * 10^{-3}$ m³/hour.

Acid leach solution comprises, kg/m³: Fe - 20,1; Cu - 7,2; Zn - 25,4; H₂SO₄ - 77,0. Extraction of metals in acidic leaching step, the solution was, %: Fe - 93,4; Cu -95,2; Zn- 93,5. Such solutions can be sent into circulation leach fresh portions Ceck-1, and after purification by neutralization and iron - by electrolytic extraction of copper, and after purification by cementation of copper - by electrolytic recovery of zinc.

Table 5: Composition of the products and the extraction stage the acid leaching of the Ceck-1

	Output, g	Fe	Cu	Zn	Pb	S	O ₂	H ₂	Other
Ceck-2	4,31*10 ⁻³	0,311	0,08	0,392	1,267	0,232	1,824	0	0,202
Content, %		7,225	1,853	9,108	29,418	5,374	42,33	0	4,693
Extraction, %		6,61	4,81	6,56	100	2,14	0,94	0	73,91
Metals solution	233,81*10 ⁻³	4,402	1,58	5,588	0	10,603	191,743	19,582	0,309
Content, %		1,883	0,676	2,39	0	4,535	82,009	8,375	0,132
Extraction, %		93,41	95,2	93,45	0	97,87	99,07	100,01	113,14

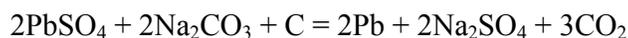
The composition of the Ceck-2 acidic leaching. Practical output Ceck was 5.68 * 10⁻³ kg, which agrees well with the calculation of the balance of the acid leaching.

Table 6: Estimated composition of Ceck-2

Fe	Cu	Zn	Pb	S	O ₂	H ₂	Other
7,225	1,853	9,108	29,418	5,374	42,33	0	4,693

According to the analysis on Cecks SPARK-1, the content of elements close to the data and makes calculations, %: Fe-8,2; Cu-3,1; Zn-10,8; Pb-32,2.

Keck-2 after the acid leaching may contain oxides of iron, copper, zinc sulfide (due to the higher oxidation start temperature during firing) and lead sulphate. Reducing the volume of the solid material from the concentrate to yield 2-caking occurred 4 times. Keck, wherein the concentrated lead and noble metals it is advisable to subject the mixture to melting with soda and a reducing agent. Since the original concentrate contained little gangue components (0, 24% SiO₂), iron oxides for slagging necessary to add silica flux. Moreover, for the recovery of lead from the most suitable conditions sulfate soda melt. Recovery may lead sulfate according to the reaction:



For this reaction, the Gibbs energy becomes negative at low temperatures: delta G273 = 17,803 kJ; delta G350 = -19,864 kJ.

Table 7: Consolidated balance firing processes, water and acid-leach processing of bulk concentrate

Received	Total, r	Fe	Cu	Zn	Pb	S	Na ₂ O	CO ₂	O ₂	N ₂	K ₂ O	Other
Concentrate	100	21,6	7,6	27,6	5,9	35,9	0	0	0	0	0	1,4
Content, %		21,6	7,6	27,6	5,9	35,9	0	0	0	0	0	1,4
Soda	109	0	0	0	0	0,687	60,517	43,306	1,036	0	2,823	0,632
Content, %		0	0	0	0	0,63	55,52	39,73	0,95	0	2,59	0,58
Air	290	0	0	0	0	0	0	0	66,7	223,3	0	0
Content, %		0	0	0	0	0	0	0	23	77	0	0
Water	1600	0	0	0	0	0	0	0	1422,24	177,76	0	0
Content, %		0	0	0	0	0	0	0	88,89	11,11	0	0
Solution H ₂ SO ₄	880	0	0	0	0	43,12	0	0	757,68	0	0	79,2
Content, %		0	0	0	0	4,9	0	0	86,1	0	0	9
Total	2979	21,6	7,6	27,6	5,9	79,707	60,517	43,306	2247,656	401,06	2,823	81,232

Cont...

Received	Total, g	Fe	Cu	Zn	Pb	S	Na ₂ O	CO ₂	O ₂	N ₂	K ₂ O	Other
Gas	458,81	0	0	0	0	0	0	42,544	14,541	401,07	0	0,655
Content, %		0	0	0	0	0	0	9,273	3,169	87,415	0	0,143
Extraction, %		0	0	0	0	0	0	98,24	0,65	100	0	0,81
Solution Na ₂ SO ₄	1574,425	0	0	0	0	31,575	60,513	0,763	1424,251	0	2,823	54,499
Content, %		0	0	0	0	2,006	3,844	0,048	90,462	0	0,179	3,462
Extraction, %		0	0	0	0	39,61	99,99	1,76	63,37	0	99,99	67,09
	18,692	1,473	0,374	1,868	5,899	1,061	0	0	7,866	0	0	0,151
Content, %		7,878	2,003	9,995	31,561	5,678	0	0	42,079	0	0	0,807
Extraction, %		6,82	4,93	6,77	99,99	1,33	0	0	0,35	0	0	0,19
Solution MeSO ₄	927,009	20,128	7,226	25,732	0	47,069	0	0	800,931	0	0	25,924
Content, %		2,171	0,779	2,776	0	5,078	0	0	86,399	0	0	2,797
Extraction, %		93,18	95,08	93,23	0	59,05	0	0	35,63	0	0	31,91
Total	2978,937	21,6	7,6	27,6	5,899	79,706	60,513	43,307	2247,588	401,07	2,823	81,23
Discrepancy	-0,063	0	0	0	-0,001	-0,001	-0,003	0,001	-0,067	0,01	0	-0,003

The calculation of material balances. Experimental results obtained during the firing №3, 5,7,15 batches of 50 * 10⁻³ kg with a degree of desulfurization of 82.9-97.6% indicate the possibility of achieving full desulfurization sulfide concentrates at relatively low temperatures of 700-750°C and acceptable residence time to practice the materials in a kiln (60-120 min), without isolation of the sulfurous gases in the atmosphere. The main condition of the firing is the presence of stoichiometric (relative sulfur) amount of soda ash and implementation process in air or oxygen-enriched atmosphere, as well as to ensure good contact with the air blast calcining material.

On the basis of experience №. 3 performance with the highest degree of desulfurization experiments with aggregated HITCH concentrate were conducted. In connection with the reduction in weight of the material in the roasting and leaching stages, the integrated processing batches of granulated batch allowed to recruit a sufficient number of Ceck-2 for the preparation of the charge scheme of soda melting and melting on hold bullion. Keck-2 laminated with a calculated amount of silica and soda ash. The charge was melted in a laboratory furnace at 1150°C, the melting duration of 30 min. As a result, smelting-2 Keck obtained crude lead and slag.

According to the author's program⁶ calculated material balances batch roasting, water and acid leaching of calcine and Ceck-1. The calculation was performed based on the compositions of starting materials and the products obtained according to the basic metal content analyzes made spectrophotometrically.

Budget calculations in the table, the outputs of products in good agreement with practical results. Removing metals in acidic solution is, %: Fe-93,2; Cu-95,1; Zn - 93,2. As a result of the melting of lead extraction in rough metal equals 96.4%

CONCLUSION

Metallurgy processes focused on monometallic selective concentrates. Preparation of bulk concentrates of 10-15% increases the recovery of valuable components in the concentrate flotation step, eliminates a number of operations of selective flotation, thickening, filtering reduces reagent consumption.

Addition of alkali metal salts containing sulfides in the charge during firing and allows melting bind sulfur in the sodium and potassium sulfates. Sodium and potassium sulfates may be made to regenerate the alkali metal salts, returned into circulation, as well as to obtain the necessary amount of sulfuric acid.

In most publications on the use of alkali metal salts of sulphide concentrates, middling's and as reagents used chlorides and hydroxides of sodium and potassium, insufficient research on the oxidation of sulphide in the atmosphere containing oxygen in addition to the charge of sodium carbonate. Technology for processing of collective concentrates using sodium carbonate (soda ash), will increase the technical and economic indicators and comprehensive utilization of raw materials, to solve the problem of disposal of sulfur.

The proposed scheme of processing of polymetallic sulphide concentrates of heavy non-ferrous metals, comprising: granulating the charge with a stoichiometric amount of soda ash; oxidative roasting of granulated batch by blowing air at a temperature of 700-750°C; Water leaching of calcine with the withdrawal of soda residue and sodium sulfate; acid leaching Ceck-1 to obtain a solution of copper sulfate, zinc and iron; reducing melt soda-2 Ceck containing lead in the form of sulphate to give crude lead and small amounts of slag.

Technology will virtually eliminate the atmosphere isolation sulfur containing gases by the binding of sulfur in the sodium sulfate, which can be used as a product or directed to the soda recovery electro dialysis methods and carbonization. Sulfide-soda-sulphate technology allows solutions to extract the copper, zinc and iron at 94-95% and also lead and precious metals in the accompanying crude metal.

The use of low-temperature (700-750°C) firing using the exothermic effect of the reactions reduces energy costs for processing polymetallic concentrates. Exception release sulfur and sulfur anhydrides, typically formed in the smelting and roasting processes of sulfide concentrates, as well as the use of evolved (in the baking step) of carbon dioxide to regenerate the sodium carbonate, is a precondition for cleaner industrial process.

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