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Flotation studies of the middling product of lead-zinc ores with preliminary sulfidizing roasting of oxidized lead and zinc compounds

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ABSTRACT

The development of combined enrichment technologies becomes more significant due to the decreasing of the processed ores quality and involvement in the processing of poor, refractory ores. The difficulty in the flotation enrichment of mixed and oxidized polymetallic ores and intermediate products lies in the effective sulfidization of the surface of oxidized minerals. The sodium sulfide reagent used in flotation does not always provide complete sulfidization of the mineral surface. On the intermediate product of lead-zinc ores related to the phase composition of mixed ores, flotation studies were carried out on the original product and after preliminary sulfidization by the pyro-metallurgical method. It was found that in the pre-sulfidizied product, the extraction of zinc increased to 17.23 %, and lead to 10.07 %, compared with the original product without pre-treatment.

Key words: lead-zinc ore, phase analysis, sulfidization, yield, content, extraction.

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Introduction

In the process of beneficiation of mixed polymetallic ores, a significant amount of intermediate products and beneficiation tailings are formed, the flotation processing of which is ineffective. At the same time, in addition to oxidized Zn and Pb compounds, a significant amount of pyrite is concentrated in the chamber flotation products [[1], [2], [3]].

The processing of large masses of ores, which are complex, hard-to-dress mineral raw materials, with the production of conditioned concentrates, in many cases, cannot be solved in the classical sequence "concentration plant - metallurgical plant". This fact naturally led to combined enrichment schemes, including operations of mechanical, pyro-metallurgical, and hydrometallurgical processing in a single technology implemented at the processing plant [4].

The use of combined enrichment technologies for the processing of refractory ores, combining the actual enrichment and mainly hydrometallurgical

(rarely pyro-metallurgical) processes in a single technological scheme of the enrichment enterprise is a general global trend.

A promising technological approach to the processing of refractory ores, which at the enrichment stage provides for the partial extraction of valuable metals into conditioned monometallic flotation concentrates, as well as additional extraction of valuable metals and substandard polymetallic intermediate products. The intermediate products mainly contain oxidized mineral particles of valuable metals that are not sufficiently contrasting in terms of technological properties. The complex intermediate substandard product is processed separately using chemical-metallurgical processes [[5], [6]].

An analysis of the practice of concentration plants shows that for additional extraction of metals from tailings rich in Zn and Pb of flotation enrichment of polymetallic ores, as well as for tailings from the processing of copper sulfide ores, flotation enrichment methods are used, with preliminary preparation of tailings for flotation [[7], [8], [9], [10]]. However, for the processing of tailings poor in Zn and Pb, flotation re-enrichment according to the flotation scheme of selective flotation is ineffective due to low recovery rates of metals [[11], [12], [13]]. Collective flotation of tailings does not give a positive result due to the presence of a high content of metal oxides [[14], [15], [16], [17]]. During flotation, most of these compounds remain in the chamber product. Even with fine grinding and high consumption of reagents during flotation, no more than 10% of zinc is extracted into the sulfide product [[18], [19], [20]].

Thus, the use of combined methods, including the sulfidation of oxidized surfaces of valuable minerals with their further flotation enrichment, is promising for the processing of oxidized and mixed refractory polymetallic ores and intermediate products [[20], [21], [22], [23]].

This work is devoted to the study of the flotation of the intermediate product of lead-zinc ores subjected to preliminary sulfidization of valuable minerals by the pyro-metallurgical method.

Materials and research methods

The object of the study was the intermediate product of lead-zinc ores. The sample cutting of the original sample is shown in Figure 1.

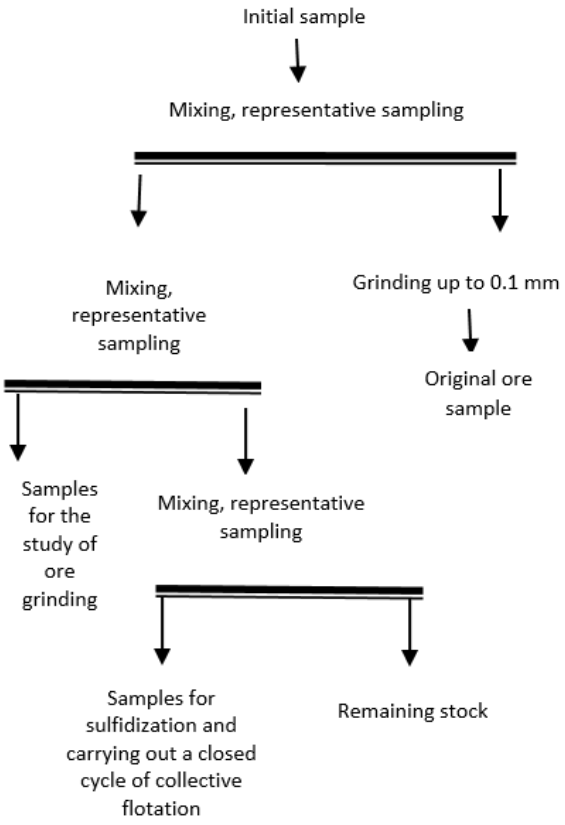

Figure 1 - Scheme of sample cutting of the initial sample

The study of the material composition of the intermediate product of lead-zinc ore: the determination of the initial content of lead and zinc was carried out by chemical analysis, the determination of the components that make up the sample by X-ray fluorescence analysis, the study of the material composition by X-ray phase analysis. Phase analyzes were performed to establish the forms of occurrence of lead and zinc minerals.

Surface sulfidization of oxidized minerals by the pyro-metallurgical method: sulfiding roasting of lead-zinc flotation tailings was carried out in a vacuum. Experiments were carried out in a fixed bed in an eclectic oven by Zhengzhou Brother

Furnace Co. Ltd (BR-17 AM-5) in a vacuum, pressure of -0.05 MPa. The holding time of the weighed samples in the oven ranged from 15 to 60 min; the temperature varied from 400 to 900 ºC. The sample was cooled under a vacuum.

Flotation enrichment: laboratory studies were carried out using the following laboratory equipment:

3) ore grinding was carried out in a laboratory ball mill at a ratio of T:W:W = 1: 0.5: 9;

4) flotation was carried out in mechanical flotation machines with chamber volumes, dm3: 1.5; 1.0; 0.5.

When conducting research on flotation, the reagent mode was used using the following reagents:

medium pH regulator - soda (Na2CO3); collectors - butyl xanthate (BKK), Aeroflot (Aero); activator - copper sulfate (CuSO4); foaming agent - T-80.

Flotation tests in a closed cycle were carried out on a sample of the original ore and a sample subjected to sulfiding roasting.

Results and discussion

After taking representative samples, chemical analysis determined the content of metals in the initial product, which amounted to 0.77% lead and 1.91% zinc. The results of X-ray fluorescence analysis of the initial sample are shown in Table 1.

Table 1 - X-ray fluorescence analysis of the original sample

Element	*Content, %*	*Element*	*Content, %*
O	51.166	Cr	0.010
Mg	0.153	Mn	0.097
Al	2.682	Fe	3.514
Si	32.048	Cu	0.076
P	0.018	Zn	1.585
S	2.515	As	0.043
Cl	0.032	Sr	0.018
K	0.583	Ba	1.487
Ca	2.991	W	0.033
Ti	0.194	Pb	0.756

From the results of X-ray fluorescence analysis, it follows that the main host elements of the sample are silicon, aluminum, calcium, sulfur, iron, and barium.

The results of studying the material composition by X-ray phase analysis are shown in Tables 2, 3 and in Figure 2.

Table 2 - Interplanar distances and phase composition of the intermediate product sample

d, Å	*I %*	*mineral*	*d, Å*	*I %*	*mineral*
4.25170	25.1	quartz	2.23549	7.1	quartz
3.34305	100.0	quartz	2.21069	6.2	quartz
3.12404	11.5	sphalerite, pyrite	2.12596	8.5	quartz
3.02886	13.1	calcite	1.97744	6.9	quartz
2.70701	8.1	pyrite	1.91221	8.4	pyrite, sphalerite
2.45524	10.3	quartz	1.81716	11.9	quartz
2.42171	6.5	pyrite	1.67099	6.6	quartz
2.27992	11.1	quartz	1.65840	5.1	quartz

Table 3 - Results of semi-quantitative X-ray phase analysis of the middling product sample

Mineral	***Formula***	***Concentration, %***
quartz	SiO_2	74.3
pyrite	FeS_2	12.5
sphalerite	ZnS	7.5
calcite	$Ca(CO_3)$	5.6

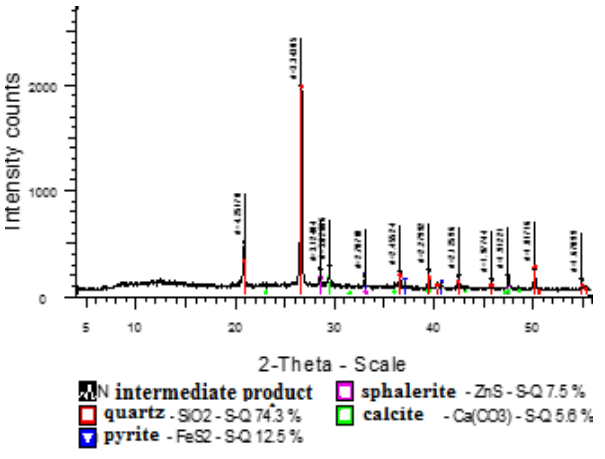

Figure 2 - X-ray diffraction pattern of an intermediate sample

It follows from the results of X-ray phase analysis that the main host minerals of the sample are quartz, pyrite, sphalerite, and calcite.

The results of phase analysis established the percentage ratio of oxidized and sulfide minerals of lead and zinc in the sample. The results of the phase analysis are shown in Table 4.

Table 4 - Results of phase analysis of lead and zinc in the sample of the intermediate product

Value	*Forms of finding / mass fraction, %*					
	Lead			*Zinc*		
	Total	*Oxidized*	*Sulfide*	*Total*	*Oxidized*	*Sulfide*
Abs	0.76	0.27	0.49	1.93	0.46	1.47
Rel	100.00	35.90	64.10	100.00	23.58	76.42

Phase analysis found that lead in the sample of the intermediate product is 64.10% sulfide, 35.90% oxidized, zinc is 76.42% sulfide, 23.58% oxidized.

Based on the results of mineralogical, phase analysis, the sample of the intermediate product belongs to the mixed type of lead-zinc ores.

Further studies were carried out on the flotation of the initial sample and the sample after sulfiding roasting.

Figure 3 shows a graph of the dependence of the content of the class minus 0.074 mm on the grinding time from the results of which it follows that in order to achieve a fineness of the crushed product of 90% class less than 0.074 mm in a laboratory mill, the time is 40 minutes.

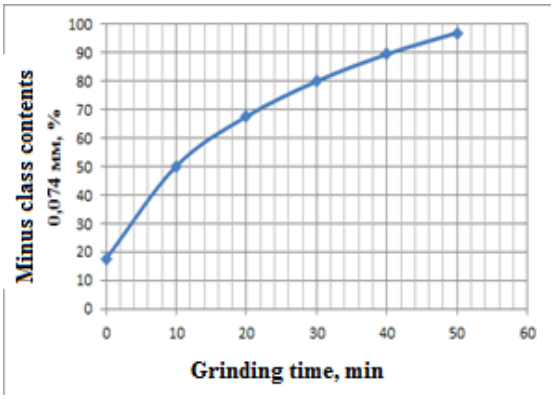

Figure 3 - Graph of class content minus 0.074 mm from grinding time for a sample of an industrial product

The consumption of reagents, flotation time and the degree of ore grinding before flotation is taken from the practice of processing similar ores and is presented in Table 5.

Table 5 - Reagent consumption, time and degree of ore grinding

The name of the operation	*Time, min*	*Reagent consumption, g/t*				
		Na_2CO_3	*БКК*	*Аэро*	*$CuSO_4$*	*Т-80*
Grinding 90% class less than 0.074 mm	40	-	-	-	-	-
Main collective flotation	12	800	150	-	-	50
Control bulk flotation	15	-	50	50	700	-
I cleaning	10	-	-	-	-	-
II cleaning	8	-	-	-	-	-

Flotation experiments were carried out according to the collective scheme shown in Figure 4 and the reagent regime shown in Table 6.

Table 6 shows the results of the experiment on collective flotation on the original sample.

Table 6 - Results of the experiment on bulk flotation on the initial sample of the intermediate product

Products	*Output, %*	*Content, %*		*Recovery, %*	
		Pb	*Zn*	*Pb*	*Zn*
Collective concentrate	12.34	2.97	11.20	47.60	72.36
Tails	87.66	0.46	0.60	52.40	27.64
Nutrition	100.00	0.77	1.91	100.00	100.00

From table 6 it follows that according to the collective scheme of flotation of the initial sample of the intermediate product, the following were obtained:

- collective concentrate with a lead content of 2.97%, zinc 11.20%, with the extraction of lead 47.60%, zinc 72.36%. The concentrate yield is 12.34%.

- tailings with a lead content of 0.46%, zinc 0.60% with a yield of 87.66%. Losses are lead 52.40%, zinc 27.64%.

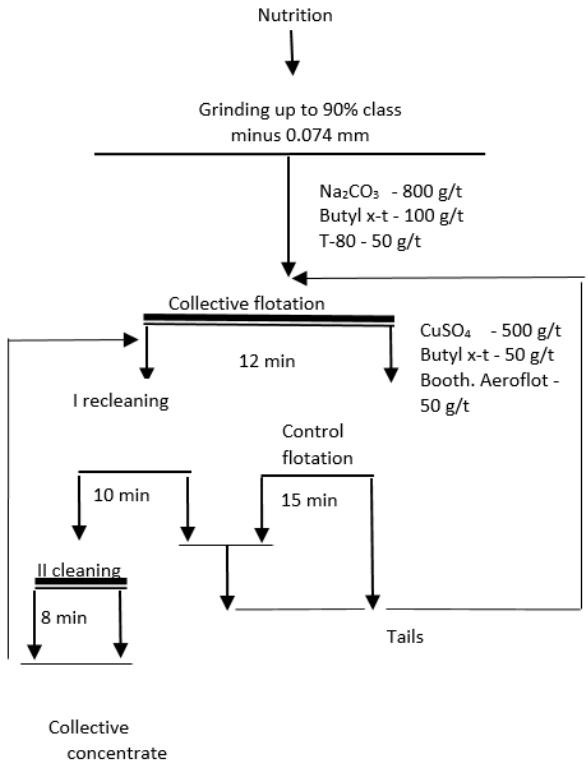

Figure 4 - Collective flotation scheme

Table 7 shows the results of a bulk flotation test on a sample of the intermediate after the sulfiding roast.

From table 7 it follows that according to the collective flotation scheme, samples of the intermediate product with preliminary sulfiding roasting were obtained:

- collective concentrate with a lead content of 3.05%, zinc 11.97%, with the extraction of lead 57.67%, zinc 89.59%. The concentrate yield is 14.37%.

- tailings with a lead content of 0.38%, zinc 0.23% with a yield of 85.63%. Losses are lead 42.33%, zinc 10.41%.

Using the method of preliminary sulfidization of the surface by the pyro-metallurgical method, the extraction of zinc into the bulk concentrate increased by 17.23%, and lead by 10.07%.

Table 7 - Results of the experiment on collective flotation on a sample of the intermediate product subjected to sulfiding roasting

Products	*Output, %*	*Content, %*		*Extraction, %*	
		Pb	*Zn*	*Pb*	*Zn*
Collective concentrate	14.37	3.05	11.97	57.67	89.59
Tails	85.63	0.38	0.23	42.33	10.41
Nutrition	100.00	0.76	1.92	100.00	100.00

Conclusion

Preliminary sulfidization of oxidized lead and zinc minerals by a pyro-metallurgical method improves the efficiency of the flotation enrichment process. The extraction of zinc into the collective concentrate increases on average by 17.23% from 72.36 to 89.59%, and lead by 10.07% from 47.60 to 57.67%.

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Қорғасын мен мырыштың тотыққан қосылыстарын алдын ала сульфидтеу арқылы күйдіре отырып қорғасын-мырыш кендерінің аралық өнімін флотациялық зерттеу

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ТҮЙІНДЕМЕ
Қайта өңделетін кендер сапасының төмендеуіне және кедей, қиын байытылатын кендерді қайта өңдеуге тартуға байланысты байытудың аралас технологияларын әзірлеу өте маңызды болып отыр. Аралас және тотыққан полиметалл кендері мен аралық өнімдерді флотациялық байыту кезіндегі қиындық тотыққан минералдардың бетін тиімді сульфидтеуге байланысты. Флотацияда қолданылатын реагент сульфидті натрий барлық жағдайда минералдың бетінің толық сульфидтенуін қамтамасыз ете алмайды. Фазалық құрамы бойынша аралас кендерге жататын қорғасын-мырыш кендерінің аралық өнімінің бастапқы өнімінде пирометаллургиялық тәсілмен алдын ала сульфидтеуден кейін флотациялық зерттеулер жүргізілді. Алдын ала сульфидтелген өнімде, өңдеуден өтпеген бастапқы өніммен салыстырғанда мырыштың алынуы 17,23%-ға, қорғасын 10,07%-ға артқаны анықталды.
Түйін сөздер: қорғасын-мырыш кені, фазалық талдау, сульфиттеу, шығын, құрылым, бөліп алу.

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Флотационные исследования промпродукта свинцово-цинковых руд с предварительным сульфидирующим обжигом окисленных соединений свинца и цинка

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АННОТАЦИЯ
В связи со снижением качества перерабатываемых руд, вовлечением в переработку бедных, труднообогатимых руд, становится более значимым разработка комбинированных технологий обогащения. Сложность при флотационном обогащении смешанных и окисленных полиметаллических руд и промежуточных продуктов заключается в эффективной сульфидизации поверхности окисленных минералов. Применяемый во флотации реагент сернистый натрий не всегда обеспечивает полную сульфидизацию поверхности минералов. На промежуточном продукте свинцово-цинковых руд относящегося по фазовому составу к смешанным рудам выполнены флотационные исследования на исходном продукте и после предварительной сульфидизации пирометаллургическим способом. Установлено, что в предварительно сульфидированный продукт извлечение цинка повысилось на 17,23%, а свинца на 10,07%, по сравнению с исходным продуктом без предварительной обработки.
Ключевые слова: свинцово-цинковая руда, фазовый анализ, сульфидизация, выход, содержание, извлечение.

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