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APPLICATION of CALCIUM POLYSULPHIDE as SULFIDIZING AGENT at FLOTATION of the OXIDIZED LEAD–BEARING ORES

Abstract: Results of studies of the Zarechnoe deposit's oxidized lead-bearing ore flotation process the with application as a sulfidizing agent of conventional sodium sulphur and new reagent – calcium pentasulphide are presented in the article. Extent of milling initial ore was optimised (88.42 % of –0.071 mm grade). For the optimum milling mode the studies on matching of an optimized consumption of sulphidizators (sodium sulphur and calcium pentasulphide) are performed. By basic technology with application of sulphide of sodium as a sulphidizator at optimum mill (88.42 % of – 0.071 mm grade), the lead concentrate with the content of lead 64.4 % at extraction of 86.6 % was received. The content of cerussite in a concentrate was about 14.9 % at extraction of 82.1 %. At application of calcium pentasulphide as a sulphidizator the lead concentrate with the content of lead 68.0 % at extraction of 88.76 % was obtained. The content of cerussite is 16.0% at extraction of 82.17 %. Application of a new sulphidizator allows to increase extraction of lead into a concentrate from 64.4 % to 68 %. The possibility of application of calcium pentasulphide as a sulphidizator instead of conventional sodium sulphur was established.

Keywords: oxidized lead-bearing ore, milling, extraction, sodium sulphur, calcium pentasulphide, sulphidizator, lead concentrate.

Introduction. Kazakhstan is a leader among CIS countries on Zn and Pb reserves and industrial rate for Zn-Pb concentrates. It can take leading positions worldwide due to raising of Zn-Pb mineral extraction.

More complex and heavy-concentrating oxidized and mixed sulphidic oxidized ores are involved in production because of increasing needs for non-ferrous metals. Extraction of the oxidized minerals of non-ferrous metals general in current technologies. Meanwhile they represent a largest raw source of non-ferrous metals.

The considerable part of polymetallic ores of Kazakhstan deposits, refer to sulphidic ones where lead and zinc contain in galenite and sfalerite (Ridder-Sokolny, Tishinsky, Maleevsky, Zyryanovsky, Belousovsky, Berezovsky's deposits). The other part refers to oxidized or carbonate ores in the upper zones deposits containing the Zn and Pb in the form of oxides - cerussite, an anglezit, smithsonite and a calamine (Rodnikovsky's ore, Zarechny, Efimovsky, Alaygyrsky deposits).

In many cases extraction of the oxidized and mixed ores does not perform separately and they are processed together with sulphidic ones.

To raise the efficiency of flotation beneficiation of the heavy-concentration used. It is applied to oxidized polymetallic ores the preliminary selective sulphidization is the oxidized surface of the components by means of hydrogen sulphide, sulphide and calcium pentasulphide, and also element sulphur, etc. [1,2].

Possibilities of hydrogen sulphide are well-known, but systematic studies of it is sulphidization impact on the oxidized minerals are not comprehensive. The low interest to hydrogen sulphide as to flotation reagent is understandable for many years. Toxicity, transportation limitations and gaseous medium were essential shortcomings for conditions of industries flotation. These circumstances were considered non-affordable for a long time [3-6]. Toxicity was also a limiting factor for sodium sulphide.

Outstanding performance of pentasulphides in case of flotation is caused by release of element sulphurous occluded on a mineral surface with the adsorptive covering. When element sulfur is fixed the mineral surface acquires the maximum floatation activity [7]. Sulphidization process was carried out at the milling stage before flotation.

Calcium pentasulphide use as a sulphidization instead of sulphurous of sodium in case of beneficiation of the heavy-concentration of the oxidized and mixed zinc-lead ores will allow to create an effective technology of similar ores. It will contribute to extraction output of the useful components and to their higher quality as well. Implementation of new processing technology will also allow, to improve working conditions and to increase gain in this area.

The task was to study details of flotation of the oxidized lead-bearing ore of the Zarechnoe deposit with calcium pentasulphide as sulphidation. To reduce costs the wastes of sulphur-containing bursts of metallurgical and oil production were selected as sulphidization.

Experimental Part and Results Discussion. The oxidized lead-bearing ore of the Zarechnoe deposit in its initial status was selected in capacity of model substance.

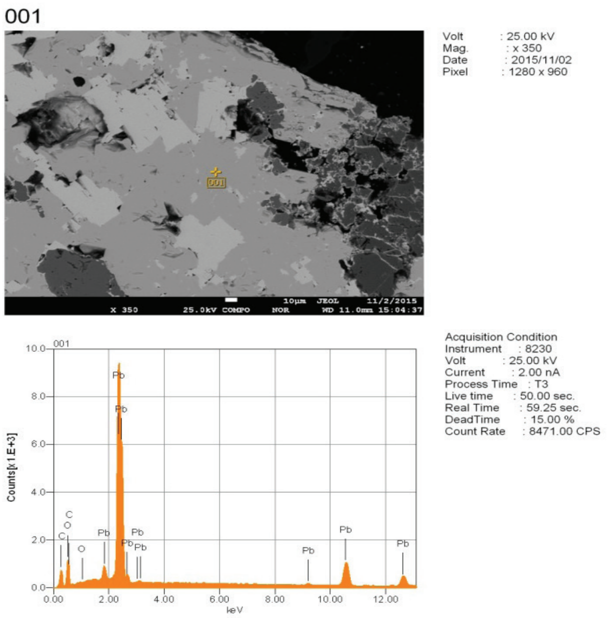
Preparation of samples included the crushing on laboratory jaw and roll cruchers and the grinding on a ball mill 40ML-000PS.

A number of research methods was used: chemical, mineralogical, X-ray phase, X-ray fluorescent, and electron probe microanalyses methods.

Lab flotation performed at laboratory flotation cells LF-290, MF-1 and FM-2 with different volumes of floatation cells - 3; 1, and 0,5 dm^3.

According to chemical analysis initial samples of the oxidized lead-bearing ore contain, %: lead – 4.8; zinc – 0.1; general iron – 0.81. The initial ore contain, %: lead – 4.8; zinc – 0.1; general iron – 0.81. phase structure is characterized by the oxidized form of 57.76-64.5 %; by sulphidic status of 25.5-32.64 %, and by plyumboyarosite of 19.0-23.02 %.

The mineralogical survey showed that initial ore contains:$PbCO_3$, PbS, $PbSO_4$, ZnS and $ZnCO_3$. Cerussite ($PbCO_3$) is observed in the form of the free grains; it splices with quartz and a galenite with grains sizes from 20 to 100 microns. Anglezit ($PbSO_4$) has preferentially pseudomorph ous form. Galenite (PbS) is observed in the form of the free grains from 14 to 200 microns. Quartz α – SiO_2, oxides and hydroxides of iron (hematite - Fe_2O_3, getit – $HFeO_2$) are in the form of the free grains or splices with quartz or mica. There are also zinc-bearing minerals – sphalerite (ZnS) and smithsonite ($ZnCO_3$) in the form of powders.



Elements	ms%	mol%	Sigma	Net	K ratio	Line
C	12.13	47.11	0.39	82666	0.0049804	K
O	12.30	35.88	0.58	204283	0.0561991	K
Pb	75.56	17.01	0.82	4951226	0.5588572	M
Total	100.00	100.00				

Figure 1 – Structure and element composition of initial oxidized lead-zinc ore

Figure 1 shows the structure and element composition of the oxidized lead-bearing ore (the Zarechnoe deposit) taken by means electron probe microanalyzer JXA-8230. There are sulphidic and oxidized lead minerals associated with dead rock in it.

This manner 4 mineral phases are found, and three other phases are belonged to non-metallic minerals. The main phase of grain is cerussite ($PbCO_3$). There are also splices of pyrites, sphalerite and galenite. The latter presents in form of splices and relic grains in cerussite (Figure 1). Sporadically it forms splices with sphalerite and non-metallic minerals. It has the following composition (%): Pb – 85.37-86.88; S – 13.12-14.63. On composition (in mass percent) cerussite corresponds to natural mineral and contains the following components with contents (%): Pb – 73.98-75.56; O – 12.24-13.0; C – 12.13-13.18.

For experiments on flotation beneficiation of oxidized lead-bearing ore (Zarechnoe deposit) the differential flow sheet was applied as a basis. The lead cycle consisted of the rough and scavenges lead flotation and two cleanings of the lead concentrate. At the grinding stage sulphuous sodium was used as sulphidizator. During the rough and scavenge lead flotations there were used the collector (butyl xanthogenate of sodium), and frother (T-80). The flotation flow sheet and the reagent mode are shown in Figure 2.

The similar experiments were performed for oxidized ore without sulphidizator. The degree of fineness for grade grinding was controlled on – 0.071mm.

According to the basic flow sheet without sulphidizator there were obtained:

- after grinding of 73.68 % the lead concentrate with the content of Pb 64.4 % at 56.01 % extraction output;
- after grinding of 88.42 % the lead concentrate with the content of Pb of 64.7 % at 55,57 % extraction output;
- after grinding of 97.48 % the lead concentrate with the content of Pb of 67.5 % at 55.27 % extraction output.

Thus, experiments without sulphidizator demonstrate that for more fine grinding quality of the lead concentrate is higher. It is consistent with fact that lead is more hydrophobic compared to other metals. Nevertheless extraction output of lead decreases to smaller values with further grinding to finest particles.

The next experiments on selection of optimal consumption of basic sulphidizator (sulphurous sodium)

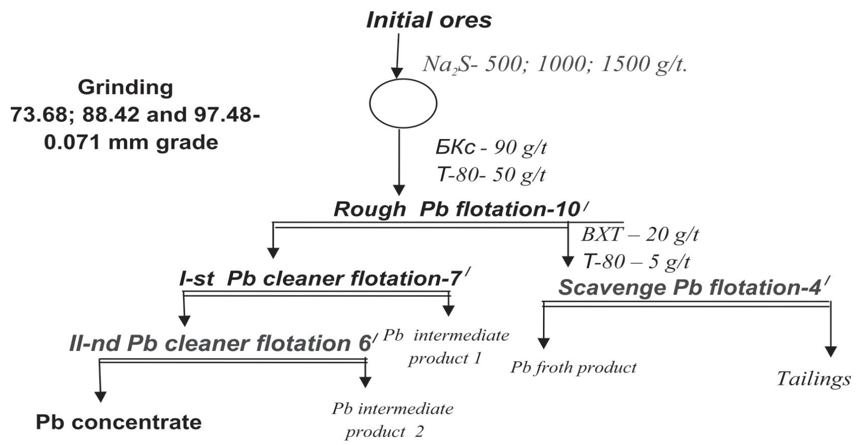


Figure 2 – The sulphid lead ore cycle flotation flow sheet ore of the Zarechnoe deposit

in a lead cycle of flotation were conducted at a different grinding of ore (73.68 %, 88.42 % and 97.48 %). The consumption of sulphurous sodium varied from 500 to 1500 g/t. It was determined that optimal consumption of sulphurous sodium for the selection lead flotation is 1000 g/t and, the results are present in Table 1.

Table 1– Extraction values of lead and cerussite from lead concentrate in case of various consumption of sulphurous sodium for various fineness of a grinding

Grinding, %	Consumption Na_2S, g/t	Content, %		Recovery, %	
		Pb	$PbCO_3$	Pb	$PbCO_3$
73,68	500	59,5	13,0	82,16	72,46
	1000	*63,4*	*13,68*	*80,29*	*74,93*
	1500	66,6	14,18	78,65	64,58
88,42	500	61,6	17,0	79,95	72,7
	1000	*64,4*	*14,9*	*86,66*	*82,10*
	1500	67,6	16,5	83,91	79,99
97,48	500	49,2	10,04	79,54	62,22
	1000	*64,5*	*19,84*	*80,76*	*77,74*
	1500	64,2	16,4	87,67	91,71

The comparative results of floatation beneficiation of the oxidized lead-bearing ore (Zarechnoe deposit) for various fineness of a grinding are in Table 2.

Table 2 – Comparative results for flotation beneficiation experiments on the oxidized lead-bearing ore (Zarechnoe deposit)

Proces-sing data	Yield, %	Content, %			Recovery, %			Grinding class –0,071 mm,%
		Pb	$PbSO_4$	$PbCO_3$	Pb	$PbSO_4$	$PbCO_3$	
Pb con-centrate	6.8	63.4	3.37	13.68	80.29	35.52	74.93	73.68
Pb intermed. prod.2	1.6	10.6	1.29	4.39	3.15	3.19	5.65	
Pb intermed. prod.1	5.28	2.32	0.62	0.99	2.28	5.07	4.21	
Froth contr. product	1.52	4.64	0.98	1.81	1.31	2.31	2.21	
Tailing	84.8	0.82	0.41	0.19	12.95	53.89	12.97	
Initial ore	100	5.36	0.64	1.24	100	100	100	
Pb con-centrate	7.44	64.4	5.6	14.9	86.66	57.59	82.10	88.42
Pb intermed. prod.2	1.78	17.5	2.75	7.5	5.63	6.76	9.88	
Pb intermed. prod.1	4	2.58	1.02	0.47	1.86	5.64	1.39	
Froth contr. product	1.24	4.64	1.64	1.82	1.04	2.81	1.67	
Tailing	85.54	0.31	0.23	0.078	4.79	27.19	4.94	
Initial ore	100	5.52	0.72	1.35	100	100	100	
Pb con-centrate	6.95	64.5	1.39	19.84	80.76	24.38	77.74	97.48
Pb intermed. prod.2	2.46	19.3	3.44	8.35	8.55	21.35	11.5	
Pb intermed. prod.1	5.35	2.06	0.58	1.01	1.98	7.83	3.04	
Froth contr. product	2.45	2.8	0.75	1.47	1.23	4.63	2.03	
Tailing	82.79	0.5	0.2	0.12	7.45	41.79	5.60	
Initial ore	100	5.55	0.39	1.77	100	100	100	

Table 2 shows that due to optimal consumption of basic sulphidizator (sulphurous sodium) of 1000 g/t there were obtained:

- after grinding of 97.48 % the lead concentrate with the content of Pb 64.5 % at extraction output of 80.76 % (where the content of cerussite was about 19.84 % at extraction output of 77.74 %);
- after grinding of 88.42 % the lead concentrate with the content of Pb 64.4 % at extraction output 86.6 % (where the content of cerussite was about 14.9 % at extraction output of 82.1 %);
- after grinding of 73,68 % the lead concentrate with the content of Pb of 63.4 % at extraction output 80.29 % (where the content of cerussite was about 13.68 % at extraction output of 74.93 %). This manner it was identified that optimal ore grinding is 88.42 %.

Another group of experimental were conducted with a new sulphidizator – calcium pentasulphide. The latter is obtained by interaction of CaO calcium oxide with sulfur and water at high temperature with component mass the following ratio S:CaO:H_2O =10:5:85. Pentasulphide of calcium was applied grinding process. Experiments were formed at the optimal ore grinding 88.42 %. The consumption of pentasulphide of calcium controlled from 500 to 1500 g/t.

The results of flotation beneficiation of the oxidized lead ore (Zarechnoe deposit) for various consumption of pentasulphide of calcium are shown in Table 3.

Table 3 – Results for flotation beneficiation experiments on the oxidized lead-bearing ore (Zarechnoe deposit) with different consumption of pentasulphide of calcium

Processing data	Yield, %	Content, %			Recovery, %			Notes
		Pb	$PbSO_4$	$PbCO_3$	Pb	$PbSO_4$	$PbCO_3$	
Pb concentrate	7.44	64.4	5.6	14.9	86.66	57.59	82.10	Basic regime Na_2S -1000 g/t
Pb intermed. prod.2	1.78	17.5	2.75	7.5	5.63	6.76	9.88	
Pb intermed. prod.1	4.0	2.58	1.02	0.47	1.86	5.64	1.39	
Froth contr. product	1.24	4.64	1.64	1.82	1.04	2.81	1.67	
Tailing	85.54	0.31	0.23	0.078	4.79	27.19	4.94	
Initial ore	100	5.52	0.72	1.35	100	100	100	
Pb concentrate	6.0	65.2	4.84	16.9	81.99	37.61	75.61	CaS_n -500 g/t
Pb intermed. prod.2	2.3	16	5.2	8.0	7.71	15.49	13.72	
Pb intermed. prod.1	5.6	2.6	1.59	0.77	3.05	11.53	3.21	
Froth contr. product	3.0	3.2	1.9	1.78	2.01	7.38	3.98	
Tailing	83.1	0.3	0.26	0.056	5.22	27.98	3.47	
Initial ore	100	4.77	0.77	1.34	100	100	100	
Pb concentrate	7,3	68,0	5,84	16,0	88,76	49,88	82,17	CaS_n -1000 g/t
Pb intermed. prod.2	2	15,8	4	7,43	5,655	9,36	10,45	
Pb intermed. prod.1	4	2,6	1,59	0,77	1,86	7,44	2,17	
Froth contr. product	1,3	2,9	2,2	1,76	0,67	3,35	1,61	
Tailing	85,4	0,2	0,3	0,06	3,05	29,97	3,60	
Initial ore	100	5,58	0,85	1,42	100	100	100	
Pb concentrate	6,8	70	5,62	14,8	83,98	43,5	74,49	CaS_n -1500 g/t
Pb intermed. prod.2	2,6	18	3,8	8,7	8,25	11,24	16,74	
Pb intermed. prod.1	4,3	3	1,28	0,77	2,27	6,26	2,451	
Froth contr. product	1,3	4,3	0,85	1,78	0,986	1,25	1,71	
Tailing	85	0,3	0,39	0,073	4,50	37,73	4,59	
Initial ore	100	5,66	0,875	1,35	100	100	100	

The optimal consumption of pentasulphide of calcium was about 1000 g/t this way the lead concentrate with the content of Pb 68.0 % at extraction output of 88.76 % was obtained. Content of cerussite in a lead concentrate was about 16.0 % at extraction output of 82.17 %.

The results of flotation beneficiation of the oxidized lead ore Zarechnoe deposit, that's possibility of application of pentasulphide of calcium as a sulphidizator instead of conventional sulphurous sodium was established.

Conclusions. Element and phase compositions were studied and flotation beneficiation flow sheet was developed for the oxidized lead-bearing ore from the Zarechnoe deposit. The initial ore contain, %: lead – 4.8; zinc – 0.1; general iron – 0.81. Phase structure of lead is characterized by the oxidized form of 57.76-64.5 %; by sulphidic status of 25.5-32.64 %, and by plyumboyarosite of 19.0-23.02 %.

Using the basic technology with sulphidizator (sulphurous sodium) for optimal ore grinding of 88.42 % (-0,071 mm grade) the lead concentrate with the content of Pb 64.4 % at extraction output of 86.6 % (where the content of cerussite was about 14.9 % at extraction output of 82.1 %) was obtained.

Due to application of calcium pentasulphide the lead concentrate with the content of Pb 68.0 % at extraction output of 88.76 % (where the content of cerussite in a lead concentrate was about 16.0 % at extraction output of 82.17 %) was obtained.

The optimal consumption for both sulphidizators is 1000 g/t. the experiments showed a possibility to replace a conventional sulphidizator (sulphurous sodium) with a new sulphidizator (calcium pentasulphide). This reagent is less toxic than sulphurous sodium and more safe from environmental point of view. From another hand it is oxidized quickly with sludging. It complicates sulphidization process of a of the oxidized lead minerals resulting to decrease in lead extraction output and content of lead sulphides in concentrate.

Thus, at optimal ore grinding ratio of 88.42 % (-0,071 mm grade) and, the optimal consumption of a sulphidizator (1000 g/t) the possibility to use calcium pentasulphide as a sulphidizator instead of basic sulphurous sodium is shown. The lead extraction output increases in a concentrate from 64.4 % to 68 %.

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ТҮЙІНДЕМЕ

Мақалада, Заречный кенорнындағы тотықты қорғасынқұрамды кендердің флотациясына, сульфидтегіш ретінде дәстүрлі күкіртті натрий мен жаңа реагент- кальций полисульфидің қолданғандағы зерттеу нәтижелері келтірілді. Бастапқы кеннің (88,42% -0,071 мм. класс) оңтайлы ұнтақтау дәрежесі анықталынды. Кенді ұнтақтаудың оңтайлы дәрежесі анықталғаннан кейін, арықарай сульфидтегіштер: натрий сульфиді мен кальций полисульфидінің оңтайлы шығынының зерттеу нәтижелері көрсетілді. Базалық технология бойынша, сульфидтегіш ретінде натрий сульфидің қолдана отырып оңтайлы ұнтақтау дәрежесі -0,071 мм класста 88,42% қорғасын концентраты алынды, ондағы қорғасынның үлесі 64,4 % болғандағы бөліп алу дәрежесі 86,6%, ал церусситтің үлесі 14,9%, бөліп алу дәрежесі 82,1 % болатындығы көрсетілді. Сульфидтегіш ретінде кальций полисульфидің қолдағанда, қорғасын концентратындағы қорғасынның үлесі 68 %, бөліп алу дәрежесі 88,76 %, ал ондағы церусситтің үлесі 16,0%, бөліп алу дәрежесі 82,17 % құрайды. Демек, жаңа сульфидтегішті қолдансақ, қорғасын концентратының бөліп алу дәрежесін 64,4% дан 68%-ға дейін жоғарлатады. Зерттеу нәтижелерімен, сульфидтегіш ретінде кальций полисульфиді қолданғанда, яғни дәстүрлі натрий сульфидінің орнын толығымен ауыстыра алатындағы бекітілді.

Түйінді сөздер: тотықты қорғасынқұрамды кен, ұнтақтау, бөліп алу дәрежесі, натрий сульфиді, кальций полисульфиді, сульфидтегіш, қорғасын концентраты

РЕЗЮМЕ

В статье представлены результаты исследований процесса флотации окисленной свинецсодержащей руды месторождения Заречное с использованием в качестве сульфидизатора традиционного сернистого натрия и нового реагента – полисульфида кальция. Проведен подбор оптимальной степени измельчения исходной руды (88,42 % класса -0,071 мм). При оптимальном режиме измельчения руды проведены исследования по подбору оптимального расхода сульфидизаторов: сульфида натрия и полисульфида кальция. По базовой технологии с применением сульфида натрия в качестве сульфидизатора при оптимальном измельчении 88,42 % класса –0,071 мм, получен свинцовый концентрат с содержанием свинца 64,4 % при извлечении 86,6 %. Содержание церуссита в концентрате составило 14,9 % при извлечении 82,1 %. С применением полисульфида кальция в качестве сульфидизатора получен свинцовый концентрат с содержанием свинца 68,0 % при извлечении 88,76 %. Содержание церуссита составило 16,0 % при извлечении 82,17 %. Использование нового сульфидизатора позволяет повысить извлечение свинца в концентрат с 64,4% до 68%. Исследованиями показана возможность использования полисульфида кальция в качестве сульфидизатора взамен традиционного сульфида натрия.

Ключевые слова: окисленная свинецсодержащая руда, измельчение, извлечение, сульфид натрия, полисульфид кальция, сульфидизатор, свинцовый концентрат

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